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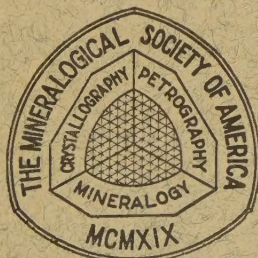
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Nos. 1 and 2

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THE LAZULITE-SCORZALITE ISOMORPHOUS SERIES¹

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ABSTRACT

The lazulite-scorzalite isomorphous series—expressed by the oxide formula $(\text{Mg}, \text{Fe}^{2+})\text{O} \cdot \text{Al}_2\text{O}_3 \cdot \text{P}_2\text{O}_5 \cdot \text{H}_2\text{O}$ —is established in this paper on the basis of chemical analyses, indices of refraction, and specific gravity of eight selected specimens. The data for six of these are new. The molecular ratio of $\text{Mg}:\text{Fe}$ in these analyses ranges from 88:12 to 24:76, with the weight per cent of MgO ranging from 11.97 to 2.93, and the weight per cent of FeO correspondingly from 2.80 to 17.06. The progressive increase in divalent iron is correlated with a progressive increase of indices of refraction, specific gravity, and birefringence. Evidence is submitted that discredits the name calcium lazulite; for the significant percentages of CaO and of SiO_2 , reported in more than two dozen analyses in the literature are believed to be the result of chemical analyses of impure samples. Geological occurrences of lazulite and scorzalite are classified into four groups and world-wide geographic occurrences are noted in accordance with this classification.

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INTRODUCTION

The lazulite-scorzalite isomorphous series—expressed by the oxide formula $(\text{Mg}, \text{Fe}'')\text{O} \cdot \text{Al}_2\text{O}_3 \cdot \text{P}_2\text{O}_5 \cdot \text{H}_2\text{O}$ —is established essentially from the data obtained by the writers on 8 selected samples representative of the series. The name lazulite has been used for over a century but the name scorzalite was only recently introduced by Pecora and Fahey (1949a) for the iron analogue of lazulite.

Most of the forty or so chemical analyses of lazulite available in the literature are not acceptable for use in this series because three rigid requirements so necessary in preparing a variation diagram were not fulfilled: (1) reliable chemical analysis on samples plus-99 per cent pure; (2) specific gravity determined on a portion of the analyzed sample; and (3) precise optical data determined on a split of the analyzed sample.

Sample selection and preparation

In the period 1945–49, several dozen lazulite specimens obtained from museums or individuals, including a number collected in the field by Pecora, were examined optically to establish the range in their indices of refraction. From these were selected eight specimens that demonstrated a representative distribution over this range.

All the samples were prepared uniformly. Handpicked, visibly pure fragments of a single specimen, or adjacent small masses in the matrix of a single specimen, were crushed so as to pass a 100-mesh screen. The powdered sample was washed free of dust by repeated decanting with water. The air-dried 100–200 or 200–300 mesh fractions were purified by means of heavy liquids (methylene iodide and acetone) and a Frantz isodynamic separator. The sample was repeatedly inspected in immersion liquids until a plus-99 per cent purity was established.

The purified sample was divided into two parts. One portion, ranging from 5 to 25 grams, was used for the chemical analyses and for determination of specific gravity with an Adams-Johnston fused silica pycnometer. The other portion was retained for precise optical studies. The indices of refraction were determined in sodium light and an Abbé refractometer was used to check the index liquids for each measurement.

Description of the selected specimens

The 8 specimens selected to demonstrate the lazulite-scorzalite isomorphous series are listed in Table 1, with general information on locality, habit and occurrence, and mineral association. Two of the specimens are from Minas Gerais, Brazil, and the others are from the United States. All the samples are now deposited with the Curator of Mineralogy, U. S. National Museum, Washington, D. C.

TABLE 1. GENERAL DESCRIPTION OF 8 SELECTED SAMPLES OF THE LAZULITE-SCORZALITE SERIES

Code Name (See Table 2)	Locality	Habit and occurrence	Associated minerals	References
1. Dattas	4 Kms. north of the Diamantina-Serro road, 15 Kms. west of Dattas, Minas Gerais, Brazil.	Irregular masses in quartz vein in sericitic quartzite (itacolomite).	Quartz, sericite, rutile.	E. R. Swoboda (personal communication)
2. Graves Mt.	Summit of Graves Mountain, Lincoln County, Ga.	Euhedral crystals in sericitic quartzite.	Quartz, kyanite, rutile, staurolite, garnet, pyrophyllite.	Shepard (1859) Watson (1912, 1921)
3. Clubbs Mt.	Southern terminus of Clubbs Mountain, Gaston County, N. C.	Irregular grains in quartz-kyanite vein in quartzite.	Same as Graves Mountain.	Hunter (1853) Smith and Brush (1853)
4. Black Eagle	Black Eagle andalusite claim, White Mountains, Mono County, Calif.	Irregular masses in quartz dike in schist.	Quartz, calcite, hematite.	Knopf (1917) C. D. Woodhouse (personal communication)
5. Breyfogle	Breyfogle Canyon, near Death Valley, Inyo County, Calif.	Irregular masses in quartz vein.	Quartz, muscovite, rutile.	Cloudman (1919) Sterrett (1911)
6. Corrego Frio	Head of Corrego Frio, near Divino, municipio of Conselheiro Pena, Minas Gerais, Brazil.	Irregular grains in border zone of granitic pegmatite.	Quartz, muscovite, albite, apatite, brazilianite, souzalite.	Pecora and Fahey (1949 a)
7. White Mt.	White Mountain andalusite mine, Mono County, Calif.	Irregular masses in quartz dike.	Quartz, andalusite, rutile, corundum, muscovite, diaspore, aluminite, pyrophyllite, augelite, woodhouseite.	Jeffrey and Woodhouse (1931) Kerr (1932) Lemmon (1937)
8. Victory	Victory mica mine, 2 miles northeast of Custer, Custer County, S. Dak.	Irregular grains in border zone of granitic pegmatite.	Quartz, muscovite, albite, tourmaline, triphylite.	Pecora and Fahey (1949 b)

The material from Graves Mountain occurs as well-formed crystals in quartzite; two other specimens are from granitic pegmatites and five from quartz-rich veins or dikes in metamorphic rocks.

DESCRIPTIVE MINERALOGY

Morphology

Well-formed crystals of lazulite occur at but a few localities. Prüfer (1847) described the crystallography of lazulite from druses in quartz veins near Werfen, Salzburg, Austria, and Shepard (1859) figured crystals recovered from quartzite at Graves Mountain, Lincoln County, Ga. About 16 forms have been reported from these localities. Only three

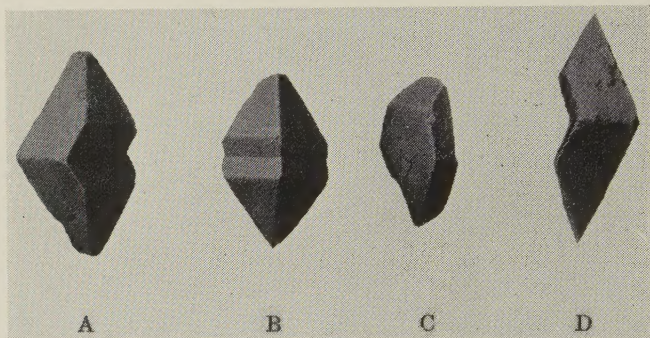


FIG. 1. Lazulite crystals from Graves Mountain, Ga. *A*, view of twinned bipyramid from the left side; *B*, twinned bipyramid from the front; *C*, tabular habit; and *D*, twinned tabular crystal from the right. (Natural size.)

forms are noted in Graves Mountain crystals— $p(111)$, $e(\bar{1}11)$, and $t(101)$. Dana (1892) and Hintze (1933) summarize the crystallography of lazulite from both localities. The twin laws noted are (223) and (100) . Baier (1941) reexamined lazulite specimens from several localities and reported several other twin laws as well.

Lacroix (1926) recorded crystals of lazulite from Madagascar as much as one centimeter in diameter and similar in habit to the Graves Mountain occurrence. Well-developed crystals are reported by Igelström (1855) as occurring in druses in vein quartz in Horrsjöberg, Wermland, Sweden.

In the Graves Mountain specimens available for this investigation, four crystal habits (see Fig. 1) are recognized: (1) bipyramids (p and e), acute or stubby, symmetrical or asymmetrical, with or without a subordinate front dome (t); (2) flat plates, representing malformed crystals with extension of one pair of pyramidal faces; (3) spindles, representing malformed crystals with extension of two pairs of pyramidal faces; and

TABLE 2. CHEMICAL ANALYSES, Mg:Fe MOLECULAR RATIOS, SPECIFIC GRAVITY, AND OPTICAL DATA FOR 8
SELECTED SAMPLES OF THE LAZULITE-SCORZALITE ISOMORPHOUS SERIES

(J. J. Fahey, analyst)

Code name in Table 1	1 Dattas, M.G.	2 Graves Mt., Ga.	3 Clubbs Mt., N.C.	4 Black Eagle, Calif.	5 Breyfogle Canyon, Calif.	6 Corrego Frio, M.G.	7 White Mt., Calif.	8 Victory, S.D.
MgO	11.97	10.38	10.02	8.42	7.98	4.23	3.38	2.93
FeO	2.80	3.95	5.22	7.96	8.94	14.74	16.27	17.06
MnO	None	None	0.03	0.01	0.14	0.11	0.07	0.10
CaO	0.08	0.06	0.06	0.08	0.13	0.02	0.08	0.03
Al ₂ O ₃	32.55	32.49	32.37	32.34	31.53	30.87	30.48	30.80
Fe ₂ O ₃	0.49	0.60	0.75	0.74	0.89	0.54	0.24	0.13
TiO ₂	0.16	0.20	0.20	0.16	0.23	0.10	0.32	0.10
P ₂ O ₅	46.12	45.79	45.42	44.97	44.31	42.90	43.31	42.67
H ₂ O +	5.90	6.48	5.83	5.72	6.27	5.86	5.46	6.10
Total	100.07	99.95	99.92	100.94	100.42	99.54	99.61	99.92
Molecular ratio Mg:Fe	88:12	82:18	77:23	65:35	61:39	34:66	27:73	24:76
Specific gravity	3.118	3.122	3.160	3.190	3.190	3.268	3.272	3.327
Index of refraction ($\pm .002$)	α	1.610	1.616	1.618	1.620	1.633	1.637	1.636
	β	1.634	1.641	1.643	1.646	1.663	1.667	1.666
	γ	1.644	1.652	1.654	1.656	1.673	1.677	1.676
2V (Calculated)	67°(—)	67°(—)	67°(—)	67°(—)	64°(—)	62°(—)	61°(—)	61°(—)

(4) bipyramids with a front reentrant representing crystals twinned on (100), or around the *c*-axis. In thin section the twin composition plane is an irregular, jagged surface essentially parallel to (001).

Most commonly lazulite and scorzalite occur as irregular masses mixed with other minerals. The mineral breaks with good cleavage along (110) and (101). As a result most of the grains in immersion lie so oriented as to give off-centered interference figures. Twin lamellae are common in specimens from most localities.

Variation in chemical composition

Chemical analyses of the 8 selected samples of lazulite and scorzalite are listed in Table 2 in the order of their increase in divalent iron. Only two of these analyses (nos. 6 and 8) have hitherto been published. The usual methods of analysis were used, except for the determination of divalent iron. The members of the lazulite-scorzalite isomorphous series are so difficultly soluble in the dilute solution of H_2SO_4 and HF used in this determination that it was necessary to grind the samples to pass a 300-mesh sieve and, further, to collect the undecomposed portion found in the beaker after titration with permanganate and carry it again through the procedure. This was repeated as many as three times for the magnesium-rich members of the series.

For the samples in Table 2, FeO ranges from 2.80 to 17.06 per cent, and MgO correspondingly ranges from 11.97 to 2.93 per cent. Inasmuch as $\text{MgO} + \text{FeO}$ makes up more than 99 per cent of the total RO, the use of the MgO:FeO molecular ratio is a reliable basis of reference on which the variation diagram of the series is established. The theoretically pure end members of the series have a composition as shown below:

Oxides	Lazulite $\text{MgAl}_2(\text{PO}_4)_2(\text{OH})_2$	Scorzalite $\text{Fe}''\text{Al}_2(\text{PO}_4)_2(\text{OH})_2$
MgO	13.34	00.00
FeO	00.00	21.57
Al_2O_3	33.74	30.54
P_2O_5	46.96	42.50
H_2O	5.96	5.39
Total	100.00	100.00

The specimens selected for special study therefore cover a wide enough range to establish reasonably the isomorphous series.

In Table 3 are listed 13 additional chemical analyses of lazulite—all obtained from published sources. These selected analyses include all that are judged to have been made from reasonably pure samples as well

TABLE 3. THIRTEEN SELECTED CHEMICAL ANALYSES OF LAZULITE SHOWING WIDE DIVERGENCE OF CHEMICAL COMPOSITION AND INADEQUATE DATA ON PHYSICAL PROPERTIES

	9	10	11	12	13	14	15	16	17	18	19	20	21
Locality	Stubachstollen, Austria	Paragatshai, Armenia	Mt. Sura-Iz, USSR	Keewatin, Canada	Chittenden, Vt.	Mt. Bity, Madagascar	Graves Mt., Ga.	Fischbach, Austria	Westana, Sweden	Gaston County, N. C.	Horsjöberg, Sweden	Stoddard, N. H.	Zermatt, Switz.
Analyst	Meixner	Shumilo	Beliakov	Hoffman	Gonyer	Pisani	Watson	Meixner	Blomstrand	Brush	Iglestrom	Gonyer	Gamper
MgO	11.83	12.10	12.54	13.84	12.38	9.19	9.08	9.87	9.05	10.04	8.58	6.45	8.64
FeO	0.69	1.30	2.08	2.09	2.59	3.95	3.99	4.58	7.82	8.17	10.55	11.58	11.91
MnO	0.01	0.03	—	—	—	—	—	0.08	0.18	—	trace	—	—
CaO	3.77	None	—	2.83	—	—	3.12	0.14	0.84	—	trace	—	—
Al ₂ O ₃	31.36	32.58	32.99	29.14	33.11	35.22	33.92	33.75	32.82	31.70	32.86	31.83	28.06
SiO ₂	4.05	—	—	—	—	—	6.05	—	—	1.07	—	—	3.04
P ₂ O ₅	42.16	46.66	46.38	46.39	46.17	45.30	38.25	45.88	43.83	43.76	42.52	45.10	42.97
H ₂ O+	6.30	6.25	6.05	6.47	6.24	5.80	5.83	6.03	5.72	5.59	5.32	5.19	5.61
Total	100.15	99.97	100.31	100.76	100.49	99.46	100.24	100.39	100.36	100.32	99.81	100.15	100.23
Mol. ratio Mg:Fe	97:3	94:6	91:9	93:7	90:10	81:19	80:20	78:22	67:33	69:31	59:41	50:50	56:44
Specific gravity	—	—	3.04	3.044	3.08	3.12	2.96	3.081	—	3.122	2.78	—	—
Index of refraction	—	1.621 1.625 1.634	1.616 — 1.634	— — —	1.612 1.634 1.643	— — —	1.604 1.633 1.642	— — —	— — —	— — —	— — —	1.634 1.659 1.668	— — —
2V	—	70°(—)	67°(—)	—	—	—	—	—	—	—	—	63°(—)	—

Notes on analyses:

No. 10. FeO₂ = 0.53; TiO₂ = 0.40; indices of refraction published as approximate values.

No. 11. Low birefringence indicates indices of refraction are approximate.

No. 13. Indices of refraction redetermined on analyzed sample: $\alpha = 1.608$, $\beta = 1.631$, $\gamma = 1.642$.No. 20. Indices of refraction redetermined on type specimen: $\alpha = 1.628$, $\beta = 1.654$, $\gamma = 1.663$; Specific gravity = 3.24.

as a number of the less reliable analyses. More than two dozen analyses of lazulite in the literature are of no value in this study because of the significant amounts of SiO_2 or CaO reported.

K. J. Murata made a spectrographic study of 5 of the samples shown in Table 2. His determination of minor elements present in these samples is given below:

	Graves Mt.	Clubbs Mt.	Breyfogle Canyon	Corrego Frio	White Mt.
.X%	Ti	Ti	Ti	Zn, Mn	Ti
.0X%	Ni, Ca, Si, V	Mn, Ca, Si, V, Ba	Mn, Ca, Ba, Sr, Si, V	Ti, Ca, Si	Cu, Mn, Ni, Ca, Si, V
.00X%	Mn, Zr, Sr, Cr	Ni, Mo, Zr, Ga, Sr	Ni, Cr	Ni, Co, Ga	Sr, Cr
.000X%	Cu, Ba	Cu, Cr	Cu	Cr	Co, Ba

Not found in any: B, Be, La, Y, Sn, Pb, Ag, Bi, Cd, Ge, In, Tl, Re, Th, U, W, Cb.

Invalidity of calcium lazulite

Watson (1921), on the basis of 3.12 per cent CaO reported in lazulite from Graves Mountain, Ga. (Table 3, no. 15), and 2.83 per cent CaO in lazulite from Kewatin, Canada (Table 3, no. 12), suggested the name *calcium lazulite* to distinguish this variety from calcium-free lazulite. Meixner (1937), who analyzed several lazulite specimens from Austrian localities, and Lemmon (1937), who studied lazulite from White Mountain, Calif., both accepted the name of calcium lazulite as valid. In 38 chemical analyses of lazulite published prior to 1948, 23 show more than 0.40 per cent CaO and the highest value is 8.20 per cent. The present writers offer the explanation that the chemical analyses showing a significant percentage of CaO were probably made on impure samples.

It is noteworthy that CaO is an insignificant constituent in all of the samples studied by the authors. The new Graves Mountain analysis (Table 2, no. 2) shows 0.02 per cent CaO , whereas the original analysis by Watson (Table 3, no. 15) shows 3.12 per cent. The White Mountain analysis (Table 2, no. 7), which shows only 0.02 per cent CaO , likewise does not support the acceptance of the name calcium lazulite used by Lemmon (1937, p. 943) for material from the same locality.

In the impure fractions recovered by purification of the samples analyzed for this paper, such calcium-bearing minerals as calcite, apatite, and garnet are included in the great number of mineral impurities identified. It is likely, therefore, that either or both mineral impurity and

analytical technique may account for those chemical analyses containing significant amounts of CaO.

Series nomenclature

The name scorzalite applies only to those members of the isomorphous series whose Fe:Mg molecular ratio exceeds 50:50. Accordingly, the only three known scorzalite localities are represented by analyses nos. 6, 7, and 8 in Table 2. In theoretical considerations, lazulite and scorzalite might be used as names for the pure end members of the series. Following the usage suggested by Schaller (1930), ferroan lazulite and magnesian scorzalite are acceptable as names covering the range for the series. Thus as examples, lazulite from Graves Mountain (Table 2, no. 2) is properly identified as ferroan lazulite ($\text{Mg}_{82}\text{:Fe}_{18}$) and scorzalite from White Mountain (Table 2, no. 7) as magnesian scorzalite ($\text{Mg}_{27}\text{:Fe}_{73}$).

Variation in specific gravity

The specific gravity of the 8 selected samples ranges from 3.118 to 3.327. The progressive increase in specific gravity for the series, shown in Fig. 2, also is correlated with a progressive increase in ferrous iron. The specific gravity of the Chittenden sample is plotted as published and that for the Stoddard sample was determined on type material furnished by T. R. Meyers.

Variation in optical properties

The indices of refraction of the 8 members of the lazulite-scorzalite isomorphous series listed in Table 2 are plotted in Fig. 2. The progressive increase in α , β , and γ is correlated with the progressive increase in divalent iron. Birefringence likewise increases from 0.034 for lazulite to 0.040 for scorzalite. As shown in Fig. 2, the value of $(\gamma-\beta)$ remains constant for the series, whereas $(\beta-\alpha)$ is larger for scorzalite. The value of $2V$ is somewhat smaller for scorzalite.

Data for only two other samples are plotted in Fig. 2. For one of these—Chittenden, Vt., (Table 3, no. 13)—the indices of refraction were redetermined on a duplicate of the analyzed sample; and for the other Stoddard, N. H., (Table 3, no. 20)—only a type specimen could be provided for reexamination. Both the published and redetermined values are listed in Table 3 (and footnote), but the latter only are plotted on Fig. 2. It is unfortunate that precise optical data are not given with the many other published analyses.

The absorption and pleochroism of lazulite and scorzalite are among the most striking found in minerals. With one exception, blue is the dominating color for Y and Z, and X is colorless. Lazulite from Stoddard is green but blue lazulite is admixed with the green material. The ab-

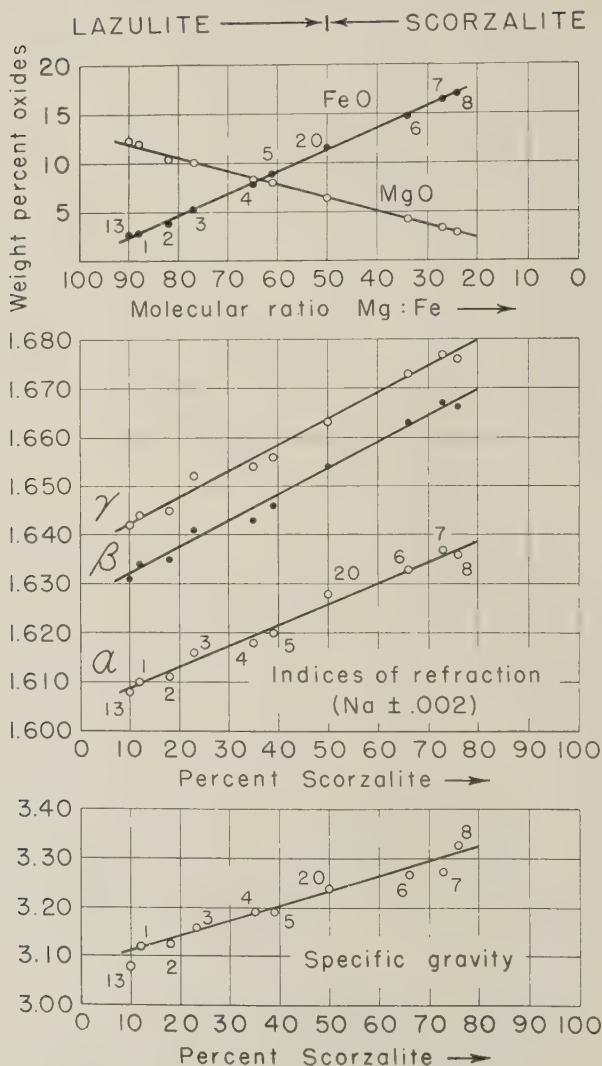


FIG. 2. Variation diagram of the lazulite-scorzalite isomorphous series. Sample numbers are identified in Tables 2 and 3.

sorption formula is the same for all specimens— $Z > Y \gg X$, but the depth of color for Z and Y varies in specimens from different localities. Lazulite from Graves Mountain, Dattas, and Clubbs Mountain is light blue, for example, and the lazulite and scorzalite specimens from California are dark blue. There appears to be no definite correlation of color with composition, although it is noteworthy that the specimens obtained from quartz veins and dikes are darker blue than those in quartzite.

The dispersion of the optic axes is $r < v$, perceptible. The orientation, established on oriented thin sections, is: Monoclinic, $Y = b$; X near c . Cleavage is good, (110) and (101).

X-ray data

X-ray powder diffraction patterns of the eight samples in Table 2 are similar, and each is identified readily as a member of the isomorphous series. Detailed measurements by J. M. Axelrod of the d -spacings (filtered Cu radiation) of two samples are given below:

Lazulite (Graves Mt.)		Scorzalite (Corrego Frio)	
d	Intensity	d	Intensity
6.16	w	6.17	m
4.72	m	4.72	m
3.60	vvw	3.60	w
3.23 (broad)	s	3.24	vs
		3.20	vs
3.14	m	3.14	s
3.07	m	3.08	m
2.55	m	2.55	m
2.34	vw	2.34	vw
2.26	w	2.26	m
2.22	w	2.22	mw
2.05	vw	2.05	vw
2.01	w	2.01	m
1.977 (broad)	mw	1.989	mw
		1.973	m
		1.828	vw
1.807	w	1.811	mw
1.787	vw	1.787	w
1.684	vw	1.682	vw
1.618	w	1.622	mw
1.600	w	1.601	w
1.568 (broad)	m	1.574 (a group)	m
1.538	w	1.541	mw
1.407	w	1.412	w
1.389	vw	1.390	vw
1.274	m	1.277	m
1.185	vw	1.186	vw
1.113	vvw	1.115	vw
.8315 (α_1)	vvw	.8318 (α_1)	vw
.8270 (α_1)	vw	.8294 (α_1)	vw
.7836 (α_1)	vw	.7849 (α_1)	vw
.7739 (α_1)	vw	.7746 (α_1)	vw

Note: $\lambda = 1.5418$ (α_{Ni}) and 1.5405 (α_1).

The 0.78 Å line in the patterns of all the samples shows a small variation of spacing with divalent iron content, as shown below:

<i>Sample</i>	<i>Per cent FeO</i>	<i>d(Å)</i>
Graves Mt.	3.95	0.7836
Breyfogle	8.94	.7844
Stoddard	11.58	.7846
Corrego Frio	14.74	.7849
White Mt.	16.27	.7856
Victory	17.06	.7853

The total variation is 0.0020. Interplanar spacings could be used, therefore, to place a member of the series approximately. Spacing alone would not give a reliable placement.

Berry (1948) calculated the unit cell for lazulite and scorzalite from cleavage pieces as follows:

<i>Locality</i>	<i>Unit cell, in Å</i>			
	<i>a</i>	<i>b</i>	<i>c</i>	<i>β</i>
1. Werfen, Salzburg	7.12	7.24	7.10	118°55'
2. Graves Mt., Ga.	7.14	7.27	7.16	119°18'
3. Churchill River, Canada	7.16	7.25	7.14	118°47'
4. Corrego Frio, Minas Gerais	7.15	7.32	7.14	119°00'

On the basis of his studies, Berry calculated the theoretical specific gravity of pure lazulite to be 3.14 and pure scorzalite to be 3.39. In comparison with the data plotted in Fig. 2, both of his values are higher than those obtained by extrapolation of the measured values.

OCCURRENCE

General association

Lazulite and scorzalite occur in many regions throughout the world where metamorphic rocks are intruded by granitic rocks. The age of these petrogenetic provinces ranges from pre-Cambrian to Cenozoic. The metamorphic rocks commonly exhibit a high-grade facies of metamorphism and contain veins, dikes, and replacement masses genetically related to the granitic rocks of the province.

The phosphate minerals intimately associated with lazulite or scorzalite in these regions include apatite, triphylite, wagnerite, berlinite, svanbergite, tetragophosphite, attakolite, kirrolite, trolleite, augelite, brazilianite, souzalite, and other unidentified phosphates. Admixed aluminum-rich minerals include kyanite, andalusite, sillimanite, corundum, pyrophyllite, spinel, dumortierite, and garnet. Other associated minerals are

quartz, albite, microcline, muscovite, beryl, tourmaline, rutile, ilmenite, hematite, magnetite, pyrite, chalcopyrite, calcite, and aragonite.

Lazulite or scorzalite is a common accessory mineral in several commercially significant nonmetallic mineral deposits. Among these are: (1) mica deposits in the United States, Brazil, and eastern Siberia; (2) andalusite deposits in California and Armenia; (3) diamond and gold placers in Brazil; (4) sapphire placers in the Himalaya Mountains; and (5) quartz crystal deposits in the Ural Mountains.

On the basis of information in the geological literature and field observations of Pecora, lazulite-scorzalite occurrences can be conveniently classified into four geological classes:

Class 1. Euhedra and subhedra in quartzite and associated metamorphic rocks.

Class 2. Anhedra in quartz-rich veins, dikes, and related replacement deposits in metamorphic rocks. Euhedra in druses are rare.

Class 3. Anhedra in granite pegmatites.

Class 4. Abraded fragments in alluvium or colluvium.

These occurrences are described below with representative localities for each group listed in Table 4.

Occurrence in quartzite (See Table 4, column 1)

The largest quantity of lazulite occurs in quartzite and quartzitic schist as individual or bunched grains scattered throughout the rock or aligned along quartzitic horizons in a sequence of foliated rocks. Aluminum-rich silicates and titanium-rich oxides are common mineral associates in this environment. The quartzite formations in many regions contain hematite-rich beds. In Madagascar, the lazulite-bearing quartzite beds are interlayered with dumortierite-rich schist.

Occurrence in quartz-rich veins and dikes (See Table 4, column 2)

Quartz-rich veins and dikes containing lazulite or scorzalite occur in the same metamorphic rocks described in Class 1, above, as well as in schist and gneiss of other regions. One dike, in Breyfogle Canyon, Calif., is reported to be several miles long, and another, at the White Mountain andalusite mine, is reported to be 300 feet thick. Most veins and dikes are much smaller. The lazulite or scorzalite occurs as irregular masses in the quartz or, rarely, as faceted crystals in druses. The quartz is of the massive or comb variety and clear or milky. Andalusite, rutile, kyanite, muscovite, and pyrophyllite are common mineral associates. Many rare phosphate minerals occur in the veins and dikes, particularly in Austria, Sweden, and California. The druses in the veins in Werfen, Austria, have provided many fine crystals of lazulite.

TABLE 4. GEOLOGIC AND GEOGRAPHIC OCCURRENCES OF LAZULITE AND SCORZALITE

	Class 1 Quartzite and quartzitic schist	Class 2 Quartz veins and dikes	Class 3 Granitic pegmatites	Class 4 Alluvium and Colluvium
United States	<ol style="list-style-type: none"> 1. Graves Mountain, Lincoln County, Ga. (Shepard, 1859; Watson, 1912 and 1921). 2. Crowders Mountain and Clubbs Mountain, Gaston County, N. C. (Hunter, 1853; Smith and Brush, 1853; Genth, 1873). 	<ol style="list-style-type: none"> 1. Near Chittenden, Vt. (Palache and Gonyer, 1930). 2. Near Stoddard, N. H. (Meyers, 1948) 3. White Mountains, Mono County, Calif. (Knopf, 1917; Kerr, 1932; Lenmon, 1937; Jeffrey and Woodhouse, 1931). 4. Breyfogle Canyon, near Death Valley, Inyo County, Calif. (Eakle, 1914; Sterrett, 1911). 5. Several localities in California in Madeira, Alpine, and Kern Counties. 6. In fault zone, Secs. 15 and 22, T. 37 S., R. 3 W., Grants Pass Quadrangle, southwestern Oregon. (F. G. Wells, pers. communication). 	<ol style="list-style-type: none"> 1. Numerous pegmatites in western N. H. 2. Victory pegmatite, near Custer, S. Dak. (Pecora and Fahey, 1949b). 	Boulder in bed of stream near Chittenden, Vt. (Palache and Gonyer, 1930).
Austria		In Salzburg, near Werfen, Stubach, Huttal, and Tauerns; and in Steiermark near Krieglach and Krumbach (Cornelius, 1931, 1936; Gampert, 1878; Hegenmann, 1927; Prüfer, 1845; Rammelsberg, 1845; Sigmund, 1909.)		
Belgian Congo				With kyanite in diamond-bearing river gravels in Moko district, Haut Vele. (Buttenbach, 1929).
Brazil	In sericitic quartzite near Diamantina and Dattas, Minas Gerais.	In quartzite near Diamantina and Dattas, Minas Gerais.	Corrego Frio, near Divino, município of Conselheiro Pena, Minas Gerais (Pecora and Fahey, 1948a).	In diamond placers of the Parana River near Dattas, Minas Gerais (Ferraz, 1929) and of the Paraguassú River, Bahia (Hussak, 1899).

	Class 1 Quartzite and quartzitic schist	Class 2 Quartz veins and dikes	Class 3 Granitic pegmatites	Class 4 Alluvium and Colluvium
Canada		1. In Silurian (?) quartzite near mouth of Churchill River, Keewatin. (Bell, 1878-9; and Hoffman 1878-9). 2. Mistassini, Quebec. (Hoffmann, 1890-1).		
French Equatorial Africa	In sericitic quartzite with kyanite, in Chari. (Chevalier, 1907, p. 689).			
India				In sapphire placers, Himalayan Mts. (Latouche, 1890).
Madagascar	1. Near Ankofa, Western High Plateau, with dumortierite schist. (Lacroix, 1926). 2. Near Ialamaza, southeast of Betafo (Lacroix, 1912).	In Vakonankaratra, Mount Bity district. (Lacroix, 1902, 1908, and 1912).		In surface float, Mount Bity district (Lacroix, 1908).
Portugal		In quartzite and schist of the Serra do Roberedo, northern Portugal, (Schreiter, 1929).		
Sweden	In iron-bearing quartzites of Westtanå (Blomstrand, 1868).	1. Near Horsesjöberg, Elfdalsh district, Wermland (Igelstrom, 1855). 2. In Westanå (Blomstrand, 1868).		
Switzerland		Near Zermatt, (Gampert, 1875; and Kennigott, 1866).		
U.S.S.R.	With andalusite in sericitic quartzite near Kazakhstan (Beliakov, 1945).	1. In the Paragachai andalusite deposit, near Kapudziok, Kongur-Alanghez Range, Armenia. (Movsesyan, 1939, 1941). 2. In quartz vein in schist, Mount Surai, Near-Polar Urals. (Beliakov, 1945).	Mica-bearing pegmatites in eastern Siberia. (Beliakov, 1945).	

Occurrence in granitic pegmatites (See Table 4, column 3)

Lazulite or scorzalite in granitic pegmatites selectively occurs in the border zone rather than in the central zone. This spatial relationship has been already described by Pecora and Fahey (1948 *a* and 1948 *b*). A number of other phosphate minerals, extremely sodic plagioclase, quartz, muscovite, tourmaline, and beryl are the common mineral associates in this environment. Within regions containing pegmatites, lazulite or scorzalite is found only in those districts where other phosphate minerals are abundant. A number of specimens collected from several New Hampshire pegmatites are ferroan lazulite in composition.

Occurrence in alluvium and colluvium (See Table 4, column 4)

Fragments composed of quartz and lazulite have been recovered in the diamond-bearing colluvium near Diamantina, Minas Gerais, and the gem tourmaline district of Mount Bity, Madagascar. Grains of lazulite have been identified as one of the heavy mineral associates in diamond and gold placers in Bahia, Brazil, and Belgian Congo, and in the sapphire placers in northern India.

GEM LAZULITE

The lazulite recovered in many localities, particularly in association with clear quartz, is of such clarity that it has been cut into gem stones. Kerr (1926) assigned the name *berkeyite* to gem lazulite, and this variety name can apply to gem scorzalite as well.

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AGE OF URANINITE FROM A PEGMATITE NEAR SINGAR, GAYA DISTRICT, INDIA

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WITH AN ISOTOPIC ANALYSIS OF LEAD

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ABSTRACT

The age of an analysed specimen of uraninite from one of the pegmatites of the mica-belt of Bihar, India, is rediscussed in the light of the isotopic constitution of lead separated from the residue of the analysed powder. The data are found to be insufficient to yield an unambiguous solution and the age cannot be stated more closely than 955 ± 40 m.y. The evidence is ample, however, to prove that the Bihar pegmatites, representing the end-stage of the Satpura orogenic cycle, are older than those of the Delhi cycle, already dated by a uraninite from Rajputana of which the age is 735 m.y. It is shown from tectonic considerations, combined with the above age estimates and tentatively supported by less reliable indications from the radioactive minerals of the Nellore district, that the sequence of the main Pre-Cambrian orogenic belts of India is: (1) Delhi; (2) Satpura; (3) Eastern Ghats; (4) Dharwar. The Aravalli belt is older than the Delhi and Satpura belts and may be a continuation of the Dhawar belt.

THE SATPURA OROGENIC BELT AND ITS RADIOACTIVE MINERALS

The mica-pegmatite swarm of Bihar, north-west of Calcutta, celebrated as the world's leading source of high-grade muscovite, extends for ninety miles from the Gaya district on the east to the Bhagalpur district on the west and has a width of up to twenty miles. A detailed account of the economic minerals of the area has recently been given by Dunn (1942). The pre-Gondwana rocks, which occupy the greater part of the region south of the Gangetic alluvium, have the following sequence:

Vindhyan System (probably Pre-Cambrian)

Newer Dolerites (probably post-Cuddapah)

Kolhan Series

Pegmatites (including those of the mica-belt)

Granites (including the Chota Nagpur and Singhbhum Granites)

Iron Ore Series (phyllites, schists, hematite-quartzites and migmatites)

To the south the Iron Ore Series is unconformably underlain by the Gangpur Series, which is correlated with similar rocks known as the Sausar Series further west. Both these older Series are characterised by marbles and gondites and, together with the Iron Ore Series and the granites and pegmatites which accompanied and followed the folding and metamorphism, they constitute a typical "Archaean" orogenic belt. The belt, of which the general trend is E.-W. or E.N.E.-W.S.W., is indicated on the accompanying map as the Satpura Belt. Following Krishnan (1943*a*, p. 140), the name is taken from the Satpura Range,

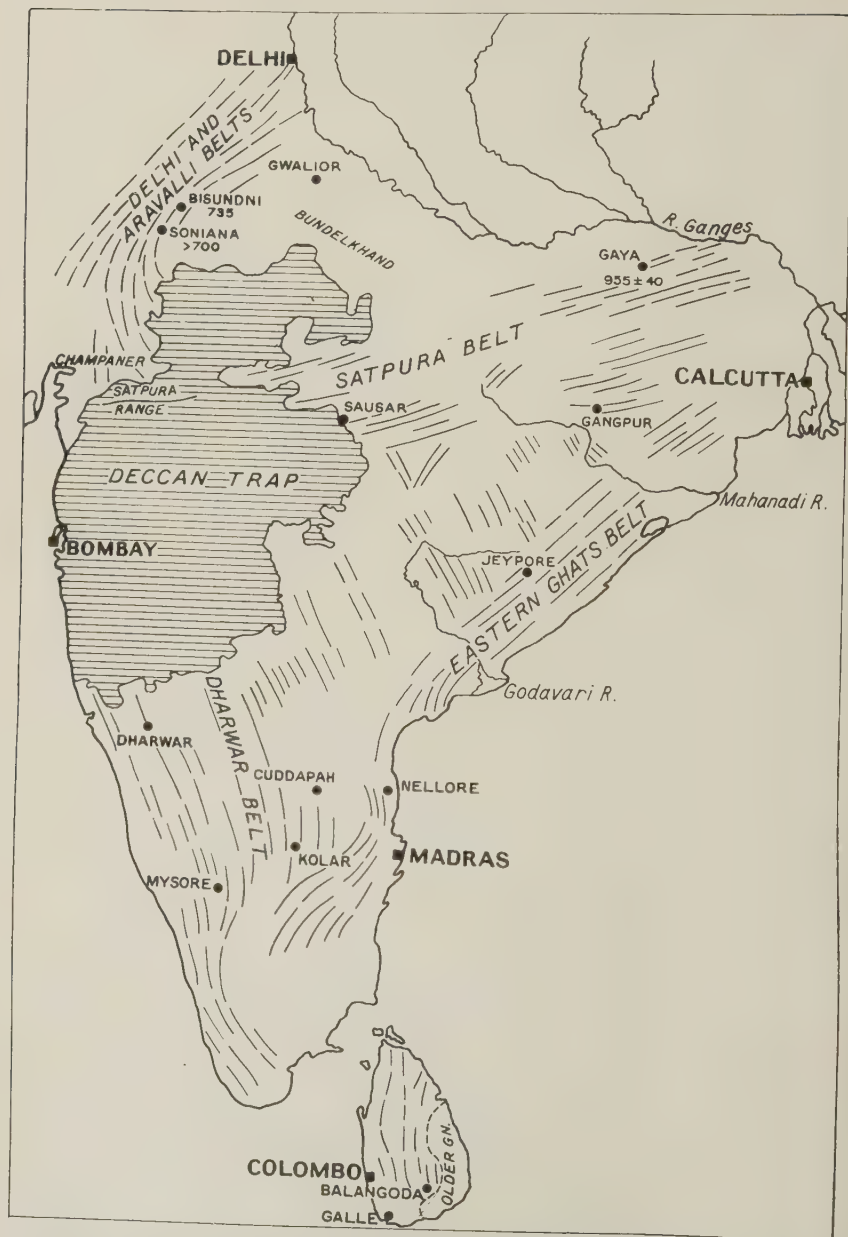


FIG. 1. Provisional tectonic map of the Pre-Cambrian orogenic belts of Peninsula India and Ceylon. The figures represent the ages of radioactive minerals in millions of years.

where schists, migmatites, granites and pegmatites have a regional trend roughly conforming to the E.N.E. direction of the range itself. Fermor (1936) regards these rocks as the geological extension, beneath the Deccan Traps, of the 'Archaean' rocks of Chota Nagpur; and Crookshank (1936, pp. 196 and 208) provisionally correlates them with the Sausar Series, apart from a group of banded hematite-quartz rocks which may be the equivalent of part of the Iron Ore Series. In the other direction the continuation of the Satpura Belt is thought to be represented by the Shillong Series of the plateau of Assam, which is separated from Bihar by the broad lowlands of the lower Ganges and Bramaputra (Krishnan, 1943*b*, p. 129).

Radioactive minerals have been found in some of the Bihar pegmatites, notably around Singar (lat. $24^{\circ}34'$: long. $85^{\circ}30'$), Pichhli (lat. $24^{\circ}36'$: long. $85^{\circ}27'$; see Tipper, 1919) and Ranchi (lat. $23^{\circ}22'$: long. $85^{\circ}20'$), and some of these have been analysed (Table 1). The apparent ages, read from the graphs prepared by Wickman (1944), in which $Pb/(U+Th)$ is plotted against $U/(U+Th)$ for various ages, range from 7830 to 980 m.y. Omitting A, the average value of the remaining four crude ages is 950 m.y.

TABLE 1. RADIOACTIVE MINERALS FROM PEGMATITES OF THE SATPURA BELT, INDIA

	Pb	U	Th	$\frac{Pb}{U+Th}$	$\frac{U}{U+Th}$	Apparent age in m.y.
A.	8.66	67.46	(? 7.0)	(.116)	(.90)	(830)
B.	8.92	64.30	8.12	.123	.89	885
C.	.49	.23	10.55	.0455	.02	965
D.	.013	0.00	.29	.0448	.00	980
E.	.021	0.00	.47	.0447	.00	980

A. Uraninite, Singar Mine, Gaya district. *Analyst*: W. R. Cripser (T. H. Holland, *Mem. Geol. Surv. India*, **34**, 1902, p. 131). Cripser did not determine ThO_2 separately, but from his analysis it is clear that ThO_2 could not have exceeded 8 per cent. Without Th the "apparent age" is 850 m.y.

B. Uraninite, Singar Mine. *Analyst*: A. Holmes (1918, p. 86 for Pb and U).

C. Monazite, Gaya district. *Analyst*: T. C. Sarkar (1941, p. 247).

D. and E. Allanite (two different crystals), Ranchi. *Analyst*: P. B. Sarkar (1946).

THE AGE OF THE SINGAR URANINITE

The "Singar Mine" quoted as the locality for the uraninites referred to in Table 1 is the Abraki Pahar mica mine mentioned by Dunn (1942, p. 237) in his account of the occurrence of uranium minerals in Bihar. Dunn writes as follows:

"At the Abraki Pahar mica mine near Singar, in Gaya district, pitchblende* was found at various times early this century. It was associated with triplite and yellow uranium ochre, the latter presumably being due to alteration of the pitchblende. According to Burton [in Hayden, 1914], the mineral occurred as nodules in a pegmatite which was 40 yards wide and 350 yards in length, but most of the pitchblende was obtained from a single pit. The largest nodule weighed 36 lbs. About 6 cwt. had been found up to 1913, and a further 16 lbs. in 1914. Rare torbernite and autunite also occurred [Tipper, 1919]."

A sample of uraninite from this locality was sent to me for age determination by the Director of the Geological Survey of India about 1914, when I was engaged in a pioneer attempt to correlate the Pre-Cambrian rocks of Mozambique with those of other countries. Only Pb and U were determined in the first place, because at that time it had not been

TABLE 2. URANINITE FROM SINGAR, GAYA DISTRICT, INDIA

CHEMICAL ANALYSIS (by A. Holmes)					
Pb	U	Th	$\frac{\text{Pb}}{\text{U}+\text{Th}}$	$\frac{\text{U}}{\text{U}+\text{Th}}$	Apparent age in m.y.
8.92	64.3	8.12	.123	.89	885
ISOTOPIC ANALYSIS OF LEAD (by W. T. Leland and A. O. Nier)					
Isotopic Proportions of Lead (Radiogenic)		Pb ²⁰⁴ *	Pb ²⁰⁶ 100	Pb ²⁰⁷ 7.20	Pb ²⁰⁸ 4.68
Per cent of Radiogenic Lead in Uraninite	}	—	7.973 RaG	.574 AcD	.373 ThD
Lead Ratios		$\frac{\text{AcD}}{\text{RaG}}$.0720	$\frac{\text{RaG}}{\text{U}}$.1240	$\frac{\text{AcD}}{\text{U}}$.0893	$\frac{\text{ThD}}{\text{Th}}$.0459
Calculated Ages (m.y.)		995	883	912	1003

* Less than 1/20,000. Original lead is therefore negligible.

realised that thorium was also a generator of lead. It was not until 1932 that an opportunity arose for determining Th. This was carried out on part of the powder originally analysed.

In the light of more recent advances it appeared highly probable that the 'age' calculated from my analysis (B, Table 1) was too low. The original analysed sample of uraninite, though free from even microscopi-

* In accordance with the distinction now generally accepted this should read "uraninite."

cally visible alteration products was penetrated by clean cracks. Some loss of radon during the lifetime of the mineral, and therefore a deficiency of RaG (Pb^{206}), would therefore seem to have been a possibility (Wickman, 1942). The consistently higher monazite and allanite "ages" (C, D and E) also suggested that my uraninite "age" was low. To attempt to clear up the doubt Professor Nier generously agreed to determine the isotopic constitution of lead separated from the residue of the analyzed sample B. I am deeply indebted to Professor Nier and Dr. Leland for their welcome collaboration and also to Mr. W. C. Hughes, Chief Analyst of the Research Department of Imperial Chemical Industries Limited at Billingham, Co. Durham, under whose supervision a sample of pure lead iodide was prepared from the mineral powder.

The results and the apparent ages calculated from them (Keevil, 1939) are given in Table 2. The 'age' corresponding to the value of AcD/RaG is read from a graph prepared by Wickman (1939).

The "spread" of the three ages calculated from the ratios involving RaG and AcD is consistent with that resulting from (a) gain of U; (b) loss of Pb; (c) loss of radon and consequent deficiency in RaG; or (d) any combination of (a), (b) and (c). These possibilities will be discussed in turn in the light of the following summary:

Type of alteration	Effect on "Ages" as calculated from			Correction required to bring all 'ages' into agreement with the 'unchanged age'
	$\frac{\text{AcD}}{\text{RaG}}$	$\frac{\text{AcD}}{\text{U}}$	$\frac{\text{RaG}}{\text{U}}$	
(a) Gain of U	unchanged* (995)	low	lower	-7.97U (per cent of mineral)
(b) Loss of Pb	unchanged* (995)	low	lower	+1.25Pb; +1.20Th
(c) Loss of Radon	high	unchanged (912)	low	+ .32RaG; + .83Th

* If the gain or loss occurred long ago, instead of recently, the 'age' from AcD/RaG would be just a trifle lower than the real age (see Holmes, Leland and Nier, 1950, Fig. 1).

(a) The assumption that the dominant effect of alteration has been a gain of U has the advantage that 995 m.y., the age that would then be the correct one, or very nearly so, is in close agreement with the age from ThD/Th and not far from the crude ages C, D and E of Table 1. However, in view of the freshness of the analysed uraninite such a large gain of U (nearly 8%) is inherently improbable and, moreover, would be un-

likely to occur without concomitant changes in Th and Pb. This solution is therefore considered to be unsatisfactory.

(b) The assumption of loss of lead implies that there has been loss of thorium-lead as well as of the uranium-leads, and when lead is arithmetically restored to bring the 'ages' based on RaG and AcD into agreement with 995 m.y. (again the correct or almost correct age) the 'age' based on ThD goes up to 1140 m.y. To reduce this to 995 m.y. involves the further assumption that 1.2% of Th has also been lost. Even so, the total losses are very much smaller than the gain required in (a) and are to that extent less improbable. The lack of visible alteration suggests the possibility that there may have been an exchange of atoms of Pb and Th for atoms of U, in which case smaller losses of Pb and Th would have been accompanied by a small gain of U. Such a combination of (a) and (b) still leaves 995 m.y. as the correct age, or nearly so.

(c) The assumption of loss of radon is not unreasonable, because such leakage is known to occur (Wickman, 1942) and would be favoured in this particular case by the physical condition of the uraninite. The maximum loss required is 4% of all the radon generated in the mineral throughout its history, corresponding to a present deficiency of .32% RaG expressed as a percentage of the mineral. In this case 912 m.y., the age from AcD/U, a ratio which is unaffected by loss of radon, becomes the correct one. There is then, however, the discrepancy of the "thorium-age" to be accounted for. To reduce 1003 m.y. to 912 m.y. implies that there was a loss of Th amounting to .83%. However, any such loss would almost certainly have been accompanied by small changes in Pb and U which would in turn have the effect of raising the correct age from 912 m.y. to something nearer 995 m.y. This would reduce the discrepancy of the "thorium age" and so reduce the amount of Th to be regarded as having been "lost."

(d) If loss of radon were the only factor concerned, then 912 m.y. could be adopted as the most probable age, whereas if no loss of radon had occurred the probable age would be 995 m.y. In the light of the above discussion it would appear that any loss of radon would also, in this case, have involved some loss of Th, accompanied, presumably, by small changes in Pb and U. The minimum total change would be one involving all the critical elements, but since there is no means of ascertaining how each one has behaved—or in what relative proportions—the evidence remains insufficient to yield an unambiguous solution.

All that can be said in conclusion is that the age of the mineral is likely to be less than 995 m.y. and more than 912 m.y. Under these circumstances the age can perhaps most fairly be stated as 955 ± 40 m.y. It had been hoped that a closer estimate would have been possible, but for this

it will be necessary to await the results of similar studies on other radioactive minerals from the Bihar pegmatites.

THE POSITION OF THE SATPURA BELT IN THE
INDIAN PRE-CAMBRIAN SEQUENCE

Although the present investigation leaves the age of the post-Satpura pegmatites still unsettled except between limits, even the lower limit of 912 m.y. suffices to indicate that the Satpura Belt represents the orogenic cycle immediately preceding that of the Delhi Belt, which extends from the neighbourhood of Delhi towards Gujerat in a general N.E.-S.W. direction (see map). Mica-pegmatites of post-Delhi age are distributed along the belt and uraninite from one of these, recently discovered by Dr. H. Crookshank, has been investigated in the same way as the Singar uraninite (Holmes, Smales, Leland and Nier, 1949).

The results, listed in Table 3, are in this case much easier to interpret. The three ages based on RaG and AcD agree as perfectly as could be expected within the limits of experimental accuracy. An age of 735 ± 5 m.y. can therefore be assigned to the mineral with confidence. The 'thorium-age' is discrepant as usual, but in this case it can only mean that there has been a trifling loss of Th (.4%). Any accompanying changes

TABLE 3. URANINITE, BISUNDNI, AJMER-MERWARA, RAJPUTANA, INDIA

CHEMICAL ANALYSIS (by A. A. Smales)					
Pb	U	Th	$\frac{\text{Pb}}{\text{U}+\text{Th}}$	$\frac{\text{U}}{\text{U}+\text{Th}}$	Apparent age in m.y.
7.95	72.9	1.4	.107	.98	730
ISOTOPIC ANALYSIS OF LEAD (by W. T. Leland and A. O. Nier)					
Isotopic Proportions of Lead (Radiogenic)	Pb^{204} <.001*	Pb^{206} 100.00	Pb^{207} 6.39	Pb^{208} .806	
Per cent of Radiogenic Lead in Uraninite	}	—	7.416 RaG	.474 AcD	.06 ThD
Lead Ratios		$\frac{\text{AcD}}{\text{RaG}}$.0639	$\frac{\text{RaG}}{\text{U}}$.1017	$\frac{\text{AcD}}{\text{U}}$.0065	$\frac{\text{ThD}}{\text{Th}}$.0429
Apparent Ages (m.y.)		740	733	733	935

* Original lead is therefore negligible.

in U and/or Pb would not affect the age calculated from AcD/RaG, i.e. 740 m.y.

The sedimentary and metamorphic formations of the Delhi cycle rest unconformably on those of the very much older Aravalli cycle. In some localities the Raialo Series intervenes between the two, this Series being provisionally correlated with the Kolhan Series, which succeeds the rocks of the Satpura Belt (Krishnan, 1943*b*, p. 135). So far as it goes this geological evidence is in accord with the geochronological proof that the Satpura Belt is older than the Delhi belt.

Inspection of the map indicates that the trend of the rocks of the Satpura Range cuts sharply across the trend of the Aravallis where the latter swing round to the S.S.E. as they approach the Deccan Traps. On this structural evidence the Satpuras appear to be younger than the Aravallis. Where the Aravallis are last seen before they disappear beneath the Deccan Traps they have a trend which is continued further south, beyond the cover of the Deccan Traps, in the rocks of the Dharwar Belt. For this reason it is provisionally supposed that the Aravallis and the Dharwars constitute parts of a single orogenic belt. This is an example of a tentative correlation that remains to be proved by the dating of radioactive minerals. It should be added that the Aravallis are underlain unconformably by a "Gneissic Complex" which represents a still older orogenic belt.

The relative positions of the Satpura and Dharwar rocks can be arrived at by another route. The map shows that the Eastern Ghats Belt, celebrated for its khondalites, kodurites and charnockites, cuts across the Dharwar Belt and therefore represents a younger orogenic cycle. The Satpura Belt, however, appears to be younger still, judging from observations made in Orissa by Krishnan (1943*a*, p. 141). Minerals suitable for dating the late pegmatites of the Eastern Ghats Belt* occur in the mica-pegmatites of the Nellore district, and already a few analyses of samarskite have been made for the purpose (Sarkar and Sen Sarma, 1946; Karunakaran and Neelakantam, 1948). The crude ages so far available (including one from a hitherto unpublished analysis by Professor Sarkar which he has kindly communicated to me) are 830, 1550 and 1760 m.y. On balance these also suggest that the Eastern Ghats Belt is older than the Satpura Belt. It is hoped that an isotopic analysis of lead prepared by Professor Sarkar from the "oldest" of these minerals will soon

* It is possible that the western margin of the Eastern Ghats Belt swings out to sea near Nellore, returning to the mainland north of Madras (*cf.* Fig. 1). If this be so, the pegmatites referred to are more likely to belong to the Dharwar Belt. It is hoped that investigation—now in progress—of an undoubted Dharwar mineral, together with further work on the Nellore minerals, will settle this question.

make possible a closer dating. There is also a possibility of dating the pegmatites from the Dharwar Belt. Ramaswamy (1943) has recorded the occurrence of monazite and samarskite from a beryl-bearing pegmatite at Yedyoor, near Bangalore. So far, however, no radioactive minerals have been recorded from the pegmatites of the Aravalli cycle.

From the evidence briefly reviewed above it appears that most of the Pre-Cambrian rocks of India fall into the following sequence. It should, however, be remembered that the supposed correlation of Aravalli and Dharwar is, as yet, no more than a plausible speculation. Moreover, it is improbable that this sequence is complete.

Vindhyan



735 m.y.

DELHI CYCLE (= ? Cuddapah)



955 \pm 40 m.y.

SATPURA CYCLE



EASTERN GHATS CYCLE



(ARAVALLI CYCLE? = ?) DHARWAR CYCLE



OLDER GNEISSIC COMPLEX (= ? Bundelkhand Gneiss)

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THREE NEW POLYMORPHS OF ZINC SULFIDE*

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ABSTRACT

Three new polymorphs of zinc sulfide are described. All are related structurally to wurtzite but differ in that they represent stacking sequences of hexagonal closest-packing of higher periodicity than that of the basic wurtzite structure. The latter structure may be designated 2H in the nomenclature of Ramsdell (1947). The new polymorphs correspond to 4H, 6H and 15R, and are isotypic with the corresponding polymorphs of silicon carbide. A tabulation of the crystallographic properties follows.

	Wurtzite-2H	Wurtzite-4H	Wurtzite-6H	Wurtzite-15R
a_0	3.811 \AA X	3.806	3.813	3.822
c_0	6.234	12.44	18.69	46.79
$a_0:c_0$	1:1.6358	1:3.268	1:4.902	1:12.242
Cell contents	Zn_2S_2	Zn_4S_4	Zn_6S_6	$\text{Zn}_{15}\text{S}_{15}$
Space group	$C6mc$	$C6mc$	$C6mc$	$R3m$

The crystals of the several polymorphs are steep pyramidal in habit and are doubly terminated with the basal pinacoid present at the analogous pole only. Parallel intergrowths were observed of 15R with 4H and of 15R with 6H. In physical properties the new polymorphs are identical with wurtzite-2H. The new polymorphs occur as tiny crystals in clay-ironstone concretions embedded at certain horizons in carbonaceous black shale of the lower Conemaugh formation over a wide area in southwestern Pennsylvania and eastern Ohio. Associated minerals are barite, sphalerite, chalcopyrite, pyrite and calcite. The concretions and the minerals therein apparently formed during the diagenesis of the sediment at essentially ordinary conditions of temperature and pressure.

INTRODUCTION

The writers recently were asked by Mr. David M. Seaman of the Carnegie Museum, Pittsburgh, to identify some tiny but perfectly de-

* Contribution from the Department of Mineralogy and Petrography, Harvard University, No. 309.

veloped hexagonal-hemimorphic crystals found by him and Mr. Howard Hamilton of Vandegrift, Pennsylvania, in clay-ironstone (siderite) concretions from near Shelocta, Pennsylvania. The crystals had the general appearance of wurtzite. Goniometric measurements on one of the crystals, however, afforded angles which deviated somewhat from those expected and it was decided to verify the tentative identification by an x -ray rotation photograph about [0001]. The photograph revealed the surprising fact that the [0001] period was exactly three times that of wurtzite. 0- and 1-layer Weissenberg photographs around the same axis proved that the lattice was hexagonal and that the length of the horizontal cell edge was identical with that of wurtzite. A study was then made of about 50 crystals available from the original specimens and from a considerable amount of additional material, in part from new localities, that had been loaned by Mr. Seaman and Mr. Hamilton. Three distinct kinds of crystals were found: (1) Crystals morphologically of dihexagonal pyramidal habit with a ρ angle of $79^{\circ} 59'$ for the dominant pyramid $\{10\bar{1}1\}$ and identical with the tripled-cell type of wurtzite mentioned above. Crystals of this type, which comprised about two-fifths of the studied material, are hereafter designated wurtzite-6H. The significance of this nomenclature, in which ordinary wurtzite is designated as 2H, is explained beyond. (2) Crystals morphologically of dihexagonal pyramidal symmetry, and very similar in habit to the preceding kind, but with a ρ angle of $75^{\circ} 9\frac{1}{2}'$ for the dominant pyramid $\{10\bar{1}1\}$. X -ray study revealed that these crystals also were based on a hexagonal lattice with the horizontal cell edge identical in length with that of ordinary wurtzite but with the [0001] period doubled. Crystals of this kind, which comprised about three-fifths of the studied material, are designated wurtzite-4H. (3) A very rare kind of crystal of ditrigonal pyramidal symmetry, with a ρ angle of $85^{\circ} 53'$ for the dominant trigonal pyramid $\{10\bar{1}1\}$. X -ray study proved the lattice to be rhombohedral, with a horizontal cell edge identical in length with those of wurtzite-2H, 4H and 6H but with a [0001] period $7\frac{1}{2}$ times that of ordinary wurtzite. This kind of crystal, represented by only four known examples, is designated wurtzite-15R. Two of these crystals proved to be parallel intergrowths of 15R with 4H and 6H.

Wurtzite-4H, 6H and 15R are found to be isotypic with the corresponding polymorphs of silicon carbide, already well known through the work of Ott (1925, 1926), Thibault (1944), Ramsdell (1947) and others. The interfacial angles of crystals of the several wurtzite polymorphs are found to agree to within a few minutes of arc with the interfacial angles of the corresponding silicon carbide polymorphs, although the absolute dimensions of the unit cells of the two groups of substances are of course dif-

ferent. The crystal structures of both the ZnS and SiC polymorphs are based on alternate closest-packed schemes of arrangement of the anions. Both the symmetry and relative dimensions of the various structures have been predicted theoretically by Belov (1939) and Zhdanov (1945) from a consideration of the possible arrangements of equally sized spheres in closest packing. The symmetry of these structures is either hexagonal or rhombohedral with the exception of one special case, the stacking sequence ABC ABC . . . , which is isometric (cubic closest-packing). Ramsdell (1947) has discussed the problem of the nomenclature of the several types of structure, and has recommended a notation comprising a letter, H or R, depending on whether the unit cell is hexagonal or rhombohedral, and a number which states the total number of hexagonally closest-packed individual layers which are stacked within the length of the unit cell. This symbol denotes the type of structure. Within each type, with the exception of the hexagonal four-layer and six-layer types, there are in addition a definite number of possible individual atomic arrangements having the same number of layers but differing in the method of stacking the individual layers. These can be described by the so-called interval sequence or zig-zag sequence of identical atoms as traced vertically in $\{11\bar{2}0\}$ sections. Types 4H and 6H each have only one stacking sequence; these have the zig-zag notation (22) and (33), respectively, and are represented by known polymorphs in both SiC and ZnS. This system is both nomenclature and classification, and is used for the substances at hand although this represents a departure from the traditional practice of applying given names to minerals.

OCCURRENCE AND ASSOCIATION

The wurtzite polymorphs occur in clay-ironstone concretions embedded in black shale of the lower Conemaugh formation of Pennsylvanian age at numerous localities over an area of at least 40 square miles in the general neighborhood of Pittsburgh, Pennsylvania. The original find was made near Shelocta, Indiana County, and eight additional localities were found shortly thereafter. The best of the known localities is in the Baltimore and Ohio Railroad cut at Wittmer, near Etna, Alleghany County. The concretions occur at levels up to twenty feet or more above the Brush Creek limestone of the lower Conemaugh formation. Further collecting activity along the outcrops of this formation will no doubt reveal many additional localities. Recently, the polymorphs have been found in the same geologic horizon near Glouster, Athens County, Ohio, about 125 miles southwest of Pittsburgh. A minor occurrence also has been found in a different horizon at the Wittmer locality, where the concretions have been found immediately above the

Pine Creek limestone which is some 60 to 90 feet over the Brush Creek member. The geology and mineralogy of the occurrences is described in more detail in an accompanying paper by Seaman and Hamilton.

The zinc sulfide polymorphs occur along fractures apparently representing shrinkage cracks in the concretions and generally are solidly embedded in platy white barite. Other associated minerals include calcite, tiny crystals of chalcopyrite and pyrite, and sphalerite both as platy masses and small tetrahedral crystals. The crystals of the polymorphs typically occur as rudely radial aggregates with the acute (antilogous) poles of the individual crystals all pointing inward. All three wurtzite polymorphs occur in immediate association, in fact often side by side or intergrown, and doubtless have crystallized simultaneously. In some instances, the isometric polymorph of zinc sulfide, sphalerite, is also directly associated and in these instances is found to have formed later than the wurtzites. Ordinary wurtzite (2H) was not observed. Both the shale containing the concretions and the Brush Creek limestone are fossiliferous and highly carbonaceous brackish water deposits. The heavy metals present in the concretions are syngenetic with the containing rock. The concretions are believed to have formed on the sea bottom or early in the diagenesis of the sediment. The development of the cracks apparently was a consequence of a process akin to the ageing and syneresis of a colloidal gel, and the concentration and crystallization of the heavy metals as the minerals noted above was a concomitant thereof. The temperatures and pressures existing at this time are presumed to have been close to normal.

PHYSICAL AND CHEMICAL PROPERTIES

The three new polymorphs are identical with each other and with ordinary wurtzite in their physical properties. The crystals average about 1 mm. in length and 0.2 mm. across. There is a distinct cleavage on $\{11\bar{2}0\}$ and traces of a cleavage on $\{0001\}$. The color is deep reddish brown to dark brown, and the fine powder is light brown. The luster is brilliant and submetallic on crystal faces and inclines toward resinous on fracture surfaces. The specific gravity of a mixed sample of the 4H and 6H polymorphs weighing 12 mg. was determined on a microbalance as 4.09 ± 0.02 . This value is practically identical with the value 4.087 reported for essentially pure wurtzite. The specific gravities calculated from the measured unit cell dimensions of the several polymorphs on the assumption that the chemical composition is ZnS are given in Table 1. The calculated values vary somewhat between each other due probably to the presence of small and variable amounts of Fe'' in substitution for Zn with a concomitant variation in cell size.

TABLE 1. X-RAY STRUCTURE CELLS OF CORRESPONDING POLYMORPHS OF ZINC SULFIDE AND SILICON CARBIDE

	Isometric		2H		4H		6H		15R	
	ZnS	SiC	ZnS	SiC	ZnS	SiC	ZnS	SiC	ZnS	SiC
a_0	5.400 kX	4.349	3.811		3.806	3.073	3.813	3.073	3.822	3.073
c_0	—	—	6.234		12.44	10.053	18.69	15.079	46.79	37.70
$a_0:c_0$	—	—	1:1.6358		1:3.268	1:3.271	1:4.902	1:4.907	1:12.242	1:12.267
Cell contents	Zn_4S_4	Si_4C_4	Zn_2S_2		Zn_4S_4	Si_4C_4	Zn_6S_6	Si_6C_6	$Zn_{16}S_{16}$	$Si_{16}C_{16}$
Space group	$F\bar{4}3m$		$C6mc$		$C6mc$		$C6mc$		$S3m$	
S.G., meas.	4.084		4.087		4.09			3.218		
S.G., calc.	4.090	3.216	4.101		4.121	3.217	4.099	3.217	4.074	3.217

Sufficient material for a quantitative chemical analysis is not yet available. Qualitative tests show the presence of large amounts of only zinc and sulfur. This fact together with the systematic geometric relations shown to ordinary wurtzite and the identity in physical properties with that substance make it virtually certain that the crystals have the composition ZnS . What doubt remains concerns the possible identity with the little-known oxysulfide of zinc, voltzite. The two available analyses of this substance, both very old, indicate the composition $\text{Zn}_5\text{S}_4\text{O}$. This formula, which can be interpreted as of an AB-type compound, and the recorded optical and physical properties suggest that voltzite may be isostructural with wurtzite. The measured specific gravity of voltzite is given as 3.66–3.80, which is considerably less than that found for the wurtzite polymorphs. The specific gravity calculated for wurtzite-4H on the basis that one-fifth of the S atoms are substituted for by O, it being assumed that there is no concomitant change in cell dimensions, is 3.43. The possibility also exists that the O substitution is serial, of the nature $\text{Zn}(\text{S}_{1-x}, \text{O}_x)$ where x is about 0.2 in the reported analyses of voltzite.

Divalent iron is known to substitute for Zn in natural wurtzite-2H up to at least $\text{Fe}:\text{Zn}=1:8$. That Fe'' is present in the material at hand is suggested by the relatively deep reddish brown color—pure ZnS is colorless—and by the fact that the crystals formed in an environment rich in ferrous iron as evidenced by the occurrence within concretions of siderite, FeCO_3 . Such a substitution may account for the small but significant variation observed in the cell dimensions and in the specific gravities calculated therefrom on the assumption that the composition is ZnS . These data are summarized in Table 1. This contrasts with the situation in SiC , where it has been shown by Thibault (1944) that the values of a_0 are exactly identical and the values of c_0 exact multiples in the several polymorphs.

DESCRIPTION OF THE POLYMORPHS

Wurtzite-2H and Sphalerite. Ordinary wurtzite is the prototype of the polymorphs here described and, although it was not identified in our material, its relevant properties may be summarized for purposes of comparison. Both natural and artificial crystals of wurtzite have been repeatedly described from a morphological point of view. Since wurtzite-2H and the 4H and 6H polymorphs belong to the same crystal class and do not differ largely in interfacial angles it might be supposed that crystals of the higher polymorphs have been mistaken in the past for the 2H modification. While several of the early morphological descriptions are not entirely satisfactory, due to the poor quality of the measured crystals, the data indicate that the crystals in all instances were of wurt-

zite-2H. It may be noted, however, that Allen, Crenshaw and Merwin (1912) obtained artificial crystals from the same solution that had very different habits, one long prismatic and another tabular on the base; these may represent different polymorphs but crystallographic data are lacking. The morphological measurements of Friedel (1866) on artificial wurtzite crystals prepared by Sidot (1866) appear to be the best of those available and lead to the ratio $a:c=1:1.6349$ in the orientation and unit of the structure cell.

The unit cell dimensions $a_0=3.811$ kX, c_0 6.234 were obtained by Fuller (1929) by the powder method on wurtzite prepared by heating pure artificial sphalerite with 10% NaCl at 1100° C. Comparable but less accurate values have been reported by Aminoff (1923) (a_0 3.80, c_0 6.23) on natural crystals from Příbram, Bohemia, which probably contained several per cent of Fe and Cd, and by Ulrich and Zachariasen (1925) (a_0 3.836, c_0 6.277) on material prepared by heating pure artificial sphalerite to 1100°. The specific gravity of wurtzite obtained by heating sphalerite (containing 0.11 per cent Fe) is given as 4.087 at 25°/4° by Allen, Crenshaw and Merwin (1912). A comparable value, 4.0648 at 25°/4°, was obtained by Madelung and Fuchs (1921) on natural material from Příbram. Other values have been reported for natural wurtzite, the highest of these being that of Breithaupt (1863), 4.072, on crystals from Příbram. The Příbram material contains small amounts of both Cd and Fe'' in substitution for Zn. The specific gravity of both wurtzite and sphalerite decreases as the Fe content increases.

The unit cell dimension of the isometric polymorph of ZnS, sphalerite, has been measured repeatedly:

a_0 5.394 kX (Gerlach, 1922)
5.395 (de Jong, 1928)
5.400 (Hartwig, 1926)
5.403 (Braekken, 1934)
5.412 (Chudoba and Mackowsky, 1939)

The best value for the pure or essentially pure compound is probably a_0 5.400 kX, which is close to the average of the reported values. The cell dimensions increase with increasing substitution of Fe for Zn, as shown by Chudoba and Mackowsky (1939) and others. The specific gravity of sphalerite containing 0.11 per cent Fe has been determined as 4.090 at 25°/4° by Allen, Crenshaw and Merwin (1912). Madelung and Fuchs (1921) give 4.0904 and 4.0946 at 25°/4° for unanalyzed natural material from Santander, Spain. On heating, pure sphalerite inverts to wurtzite at about 1020° C. The inversion temperature is lowered markedly by the substitution of Fe'' for Zn, and occurs at 880° in material with 17.06 per cent FeO.

Wurtzite-4H. When examined under moderate magnification the extreme dissymmetry of crystals of this kind is remarkable (Fig. 1). At one end is a hexagonal pyramid of high inclination which tapers to a sharp point; at the other end is the basal pedion alone, or together with a flat pyramid. On most crystals a prism truncation in zone with the pyramids

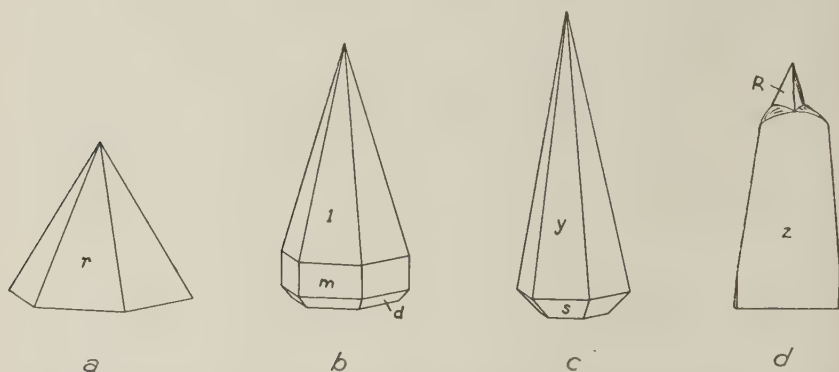


FIG. 1. Typical habit of (a) wurtzite-2H, (b) wurtzite-4H, (c) wurtzite-6H, and (d) wurtzite-15R. Forms: (a) $r\{10\bar{1}1\}$, $\bar{c}\{0001\}$; (b) $l\{10\bar{1}1\}$, $\bar{d}\{10\bar{1}3\}$, $m\{10\bar{1}0\}$, $\bar{c}\{000\bar{1}\}$; (c) $y\{10\bar{1}1\}$, $\bar{s}\{10\bar{1}3\}$, $\bar{c}\{000\bar{1}\}$; (d) $z\{10\bar{1}1\}$, $R\{01\bar{1}3\}$.

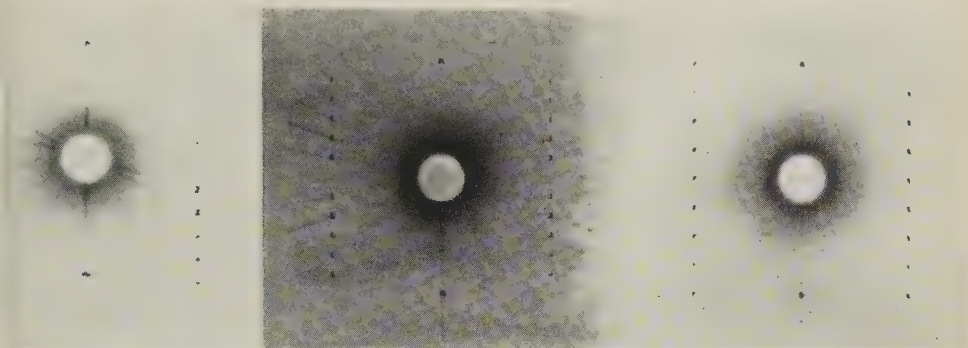
may be seen on the basal edges. The measured two-circle goniometric angles and an angle table derived in the unit and orientation of the structure cell are given in Table 2. Crystals in which the principal axis is polar are conventionally drawn with that end of the axis uppermost which acquires a positive electric charge on cooling or compression—the so-called antilogous pole. The small size of the present crystals precluded such tests and the crystals are here drawn with the steep termination uppermost, this being generally found to be the antilogous pole.

TABLE 2. ANGLE TABLE FOR WURTZITE-4H
Hexagonal—P; dihexagonal pyramidal—6mm
 $a:c=1:3.268$; $p_0:r_0=3.774:1$

Lower	Upper	Measured ρ angles*	Calculated angles			
			ϕ	ρ	M	A_2
\bar{c}	0001	—	—	0°00'	90°00'	90°00'
	m 10 $\bar{1}0$	90°00'	30°00'	90 00	60 00	90 00
\bar{d}	10 $\bar{1}3$	51 53	30 00	51 31	66 57½	90 00
	l 10 $\bar{1}1$	74 59	30 00	75 09½	61 06	90 00

* $\{10\bar{1}1\}$ average of 28 faces of medium to good quality on 7 crystals.

$\{10\bar{1}3\}$ average of 4 narrow faces of poor quality on one crystal.



Precession photographs taken in unfiltered copper radiation around the a -axis of wurtzite-4H (right), 4H (center) and wurtzite-15R (left). The spots on the vertical central lattice lines of the photographs are, (0006) and (000.15), respectively.

X-ray Weissenberg photographs were taken of the 0-levels about both horizontal axes and of the 0-level and 1-level about $[0001]$. These photographs established the lattice as hexagonal and the centrosymmetrical point group as $6/mmm$. The morphology identifies the crystal class as dihexagonal pyramidal, $6mm$. The systematic extinctions observed were $(hh2\bar{h}l)$ present only when $l=2n$, corresponding to the space group $C6mc$. The unit cell dimensions calculated from high order reflections on the 0-layer photographs are given in Table 1.

The most informative single-crystal photographs of this and of the other polymorphs of ZnS and also of SiC are afforded by the precession method as developed by Buerger (1944). Photographs taken by this method about a horizontal axis reveal the lattice type, whether hexagonal or rhombohedral, and the order number of the first reflection of $(000l)$ to appear identifies the layer period in the nomenclature of Ramsdell (1947). Reproductions of precession photographs taken around an a -axis of the 4H, 6H and 15R polymorphs of ZnS are shown in Fig. 2. The separate identity of crystals of the several polymorphs also can be done conveniently by direct comparison of Laue photographs. Drawings of such photographs taken along $[0001]$ in tungsten radiation are shown in Fig. 3. Laue photographs taken in this orientation of the SiC polymorphs have already been published by others, and Weissenberg photographs of the same substances have been reproduced by Thibault (1944) and by Ramsdell (1947).

The present polymorph is shown by its symmetry and dimensional relationships to be of the type designated as 4H, and it is isotypic with the polymorph of SiC originally designated as Type 3 and later termed 4H. While the absolute dimensions of the ZnS and SiC structures are differ-

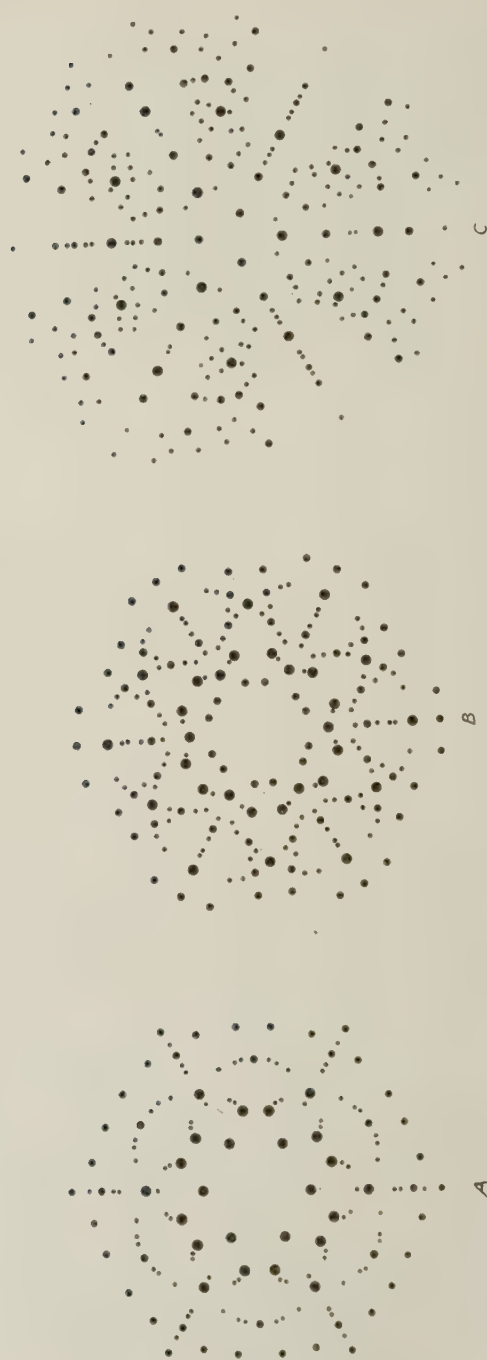


FIG. 3. Drawings of x-ray Laue photographs (W radiation at 45 KV and 10 ma) of wurtzite-4H (left), and wurtzite-6H (center) and wurtzite-15R (right). Taken along [0001].

ent, due to the difference in the size of the atoms involved, the ratios of the cell edges are practically identical and the measured interfacial angles differ by only a few minutes or so of arc. This is true not only of the 4H but also of the 6H and 15R polymorphs of ZnS and SiC. A 2H polymorph has not yet been discovered in SiC.

Wurtzite-6H. The crystal habit of this polymorph on casual examination appears to be identical with that of wurtzite-4H, with a steep upper hexagonal pyramid, a negative pedion and a flat negative pyramid. Narrow faces of additional negative pyramids are present on some crystals. Goniometric measurement, however, reveals that the dominant pyramid $\{10\bar{1}1\}$ is steeper by about 5° than in wurtzite-4H. A typical crystal habit is shown in Fig. 1. The morphological development identifies the crystal class as dihexagonal pyramidal— $6mm$. The measured two-circle goniometer angles and an angle table derived in the orientation and unit of the structure cell is given in Table 3.

TABLE 3. ANGLE TABLE FOR WURTZITE-6H
Hexagonal—P; dihexagonal pyramidal— $6mm$
 $a:c=1:4.902$; $\rho_0:r_0=5.660:1$

Lower	Upper	Measured ρ angles*	Calculated angles			
			ϕ	ρ	M	A_2
\bar{c}	0001	—	—	$0^\circ 00'$	$90^\circ 00'$	$90^\circ 00'$
\bar{o}	$10\bar{1}6$	$44^\circ 17'$	$30^\circ 00'$	43 20	69 56	90 00
\bar{n}	$10\bar{1}5$	47 21	30 00	$48\ 32\frac{1}{2}$	$67\ 59\frac{1}{2}$	90 00
\bar{s}	$10\bar{1}3$	62 40	30 00	$62\ 04\frac{1}{2}$	63 47	90 00
	y $10\bar{1}1$	79 57	30 00	79 59	$60\ 30\frac{1}{2}$	90 00

* $\{10\bar{1}1\}$ average of 78 faces of medium to good quality on 13 crystals.

$\{10\bar{1}3\}$ from one face of poor quality.

$\{10\bar{1}5\}$ average of 10 faces of poor quality on 3 crystals.

$\{10\bar{1}6\}$ average of 7 faces of very poor quality on 2 crystals.

X-ray Weissenberg photographs were taken about both horizontal axes and about the $[0001]$ axis. These established the lattice as hexagonal and the space group as $C6mc$, identical with that of wurtzite-4H and 2H. The lengths of the cell edges are given in Table 1.

Wurtzite-15R. Four hemimorphic crystals were found in the course of the study that differed from those of the two hexagonal polymorphs in possessing a marked trigonal habit without trace of the usual hexagonal pyramids. These crystals showed a steep trigonal pyramid above and a broad basal pinacoid below, the lower corners being truncated by minute faces of a negative trigonal pyramid. The upper trigonal pyramid in all

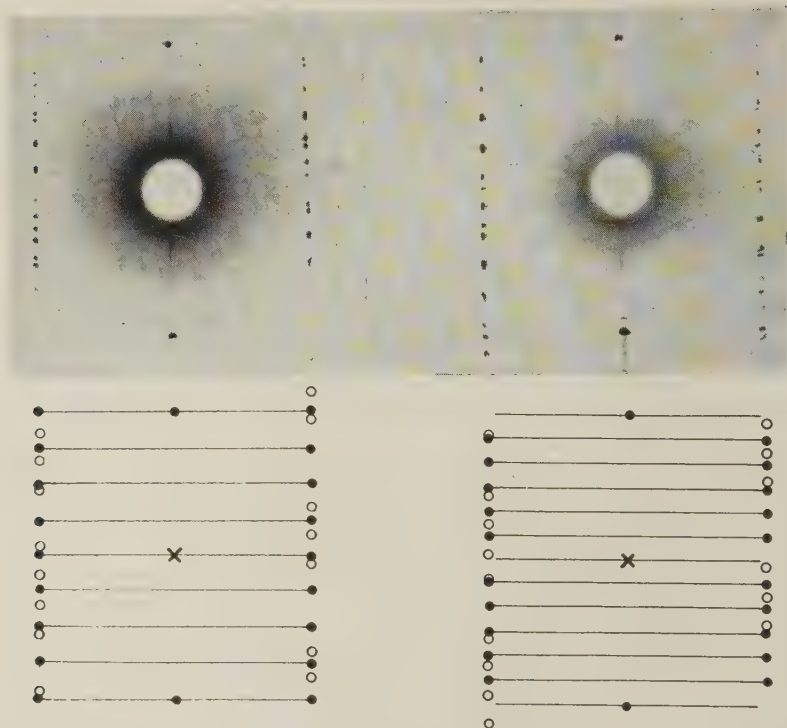


FIG. 4. X-ray precession photographs taken about an a -axis showing parallel intergrowths of wurtzite-4H and 15R (left) and wurtzite-6H and 15R (right). The x -ray beam impinging on the boundary between the intergrown parts in each case. Open circles show spots belonging to 15R, solid circles to spots belonging to 4H or 6H.

TABLE 4. ANGLE TABLE FOR WURTZITE-15R
Hexagonal—R; ditrigonal pyramidal— $3m$
 $a:c = 1:12.242$; $\alpha = 13^\circ 56'$; $p_0:r_0 = 14.136:1$; $\lambda = 119^\circ 30\frac{1}{2}'$

Lower	Upper		Measured ρ angle*	Calculated angles			
				ϕ	$\rho = C$	A_1	A_2
\bar{c}		0001 111	—	—	$0^\circ 00'$	$90^\circ 00'$	$90^\circ 00'$
	s	1.1.2.18 765	$53^\circ 52'$	$0^\circ 00'$	$53^\circ 40\frac{1}{2}'$	45 45	45 45
\bar{M}		1.0.1.13 544	47 42	30 00	47 24	50 24	90 00
	g	1014 211	74 15	30 00	74 12	$33^\circ 33\frac{1}{2}'$	90 00
	z	1011 100	85 53	30 00	85 57	30 15	90 00
\bar{h}		0.1.1.11 443	52 20	$-30^\circ 00'$	$52^\circ 06\frac{1}{2}'$	90 00	46 53
	R	0113 441	$78^\circ 03\frac{1}{2}'$	$-30^\circ 00'$	78 01	90 00	32 06

* {1011} average of 6 faces of good quality.

{1014}, {1.0.1.13} and {0.1.1.11} all from one face only.

{0113} average of 3 faces of fair quality.

{1.1.2.18} average of 3 faces of very poor quality.

four cases showed a peculiar termination consisting of a deep shelf-like indentation on the vertically trending edges rising from which was a tiny spire-like projection itself bounded by very steep positive and negative trigonal pyramids. The appearance of these crystals is shown in Fig. 1. All four crystals were identical morphologically, although all of the observed forms were not present on each individual. An angle table for the observed forms in the orientation and unit of the structure cell is given in Table 4.

A complete Weissenberg x -ray study was made of one crystal, the negative or butt end of the crystal being placed in the x -ray beam. The photographs established the lattice as rhombohedral and the centrosymmetry as $3m$. These results were confirmed by both Laue and precession photographs. The crystal class is fixed as ditrigonal pyramidal, $3m$, by the morphological development. The space group is $R3m$ as fixed by the observed characteristic reflections: $(hh\bar{2}hl)$ present when $l=3n$, $(hh\bar{0}l)$ present when $2h-l=3n$, and $(hk\bar{k}l)$ present when $h-k-l=3n$. The dimensions of the unit cell are given in Table 1. This polymorph is identified by its symmetry and dimensional relations as 15R and is isotypic with the so-called Type 1, or 15R, polymorph of SiC.

It was considered that the spire-like projection at the upper termination of the crystals might represent a different polymorph, intergrown in parallel position perhaps as a core through the main individual. Weissenberg photographs obtained by exposing only the spire in the x -ray beam, however, proved them to be identical with the main body of the crystal. Two of the crystals showed small inserted areas bounded by surface sutures, quite distinct from the spires, which proved on investigation to be different polymorphs intergrown in parallel position; these are described in the following section.

PARALLEL INTERGROWTHS BETWEEN DIFFERENT POLYMORPHS

Two crystals of wurtzite-15R were observed to have small intergrown parts irregularly bounded by surface sutures. Precession photographs were taken so that the x -ray beam impinged directly on the boundary of the intergrowth. One crystal gave a photograph consisting principally of the pattern of 15R on which was superimposed a system of spots corresponding to an a -axis precession photograph of a crystal of 6H, as shown in Fig. 4. This crystal thus is a parallel intergrowth of wurtzite-15R and 6H. The other crystal when examined similarly (Fig. 4) proved to be a parallel intergrowth of wurtzite-15R and 4H. Parallel intergrowths between different polymorphs are relatively common with SiC and have been described by Thibault (1944) and Baumhauer (1915). In SiC the surfaces of juncture are planar and parallel to $\{0001\}$ while in

wurtzite the boundaries are irregular and trend roughly vertically. In the latter instances, at least, it must be presumed that the different polymorphs grew simultaneously.

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OCCURRENCE OF POLYMORPHOUS WURTZITE IN WESTERN PENNSYLVANIA AND EASTERN OHIO*

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ABSTRACT

Three new wurtzite-type polymorphs of ZnS have been found along shrinkage cracks in clay-ironstone concretions a few feet above the Brush Creek limestone horizon of the Conemaugh Formation near Etna, Allegheny County, Pennsylvania. Two of them, wurtzite-4H and wurtzite-6H, have been noted at this horizon over an area of fifty square miles in western Pennsylvania and eastern Ohio. The third, wurtzite-15R, is reported in only four crystals from the Etna locality. The geology and mineralogy are described for the known occurrences of these polymorphs.

INTRODUCTION

Three new polymorphous forms of ZnS are described by Dr. Clifford Frondel and Dr. Charles Palache of Harvard University in the current issue of the *American Mineralogist*. It is at their suggestion that this brief accompanying paper has been prepared describing somewhat more fully the geology and mineralogy of the occurrences.

The original locality was found on a very bright afternoon, August 10, 1946, while the authors were on a fossil collecting trip to the Brush Creek limestone horizon, one half mile northwest of Shelocta, Indiana County, Pennsylvania, along U. S. Route 422. In his search for fossils the junior author happened to break open a clay-ironstone concretion from a layer about ten feet above this limestone. To his surprise, he noted some tiny brownish red crystals embedded in white, platy barite in the center of the concretion. The crystals would undoubtedly have been missed if it had not been for the brilliant, mirror-like reflections from the tiny crystal faces. The first crystals found were less than one millimeter in length and barely visible to the naked eye.

Viewing them under a microscope at the museum it was noted that they were steep pyramidal crystals with hexagonal-hemimorphic development. In the belief that they might be wurtzite, several crystals were sent to Dr. Frondel of Harvard University for verification. Wurtzite had not previously been reported from western Pennsylvania. This first polymorph proved to be wurtzite-6H of their study. Additional material from the Shelocta locality revealed another new polymorph, wurtzite-4H.

* Contribution from the Department of Mineralogy and Petrography, Harvard University, No. 310.

Subsequent collecting from a number of localities yielded a third new wurtzite polymorph at the Wittmer locality near Etna, Pennsylvania, of rhombohedral (trigonal) crystallization, wurtzite-15R. Only four specimens of this polymorph have been found, all at the Brush Creek limestone horizon at the Wittmer locality. The two hexagonal forms, wurtzite-4H and wurtzite-6H, have been found together at fifteen different localities: thirteen in western Pennsylvania and two in eastern Ohio.

GEOLOGY

The polymorphous wurtzites occur in clay-ironstone concretions in concretionary layers at various distances above the Brush Creek limestone of the lower Conemaugh Formation of the Pennsylvanian period. The concretions themselves are usually oval in shape and from two to four or five inches in length, and up to two inches in thickness with their long dimension in the plane of stratification. Sometimes the concretions form an almost unbroken layer, end to end, in black, carbonaceous shale. The Brush Creek limestone is a nodular, black, fossiliferous limestone ranging from one foot to two feet in thickness occurring some one hundred and twenty-five feet above the Upper Freeport Coal which marks the lower boundary of the Conemaugh Formation. A few tiny wurtzites of the 4H and 6H types have been found in similar concretions one foot above the Pine Creek limestone, which is also found at the Wittmer locality some ninety feet above the Brush Creek limestone.

MINERALOGY OF CONCRETIONS

The minerals of the concretions are found along shrinkage cracks. Most of the wurtzites occur in radiating groups of single crystals side by side, like small sunbursts, with the acute pyramidal terminations pointing towards the center of the radiations. The associated minerals are usually platy barite or granular calcite, in which the wurtzites are embedded. Platy and curved sphalerite is often intimately associated with the wurtzites, with crystalline pyrite and chalcopyrite common at some localities. There appears to be no particular mineral which indicates the presence of the wurtzites in the concretions other than the usually accompanying sphalerite. The wurtzites may be found alone or with any of the above mentioned minerals. If calcite is present, there is no barite, and vice versa. These two minerals are never found in the same concretions though both are present at the Wittmer locality. Here the barite-bearing concretions seem to be confined to the southern end of the cut and the calcite concretions to the center and northern end.

The size of the seam or crack is also not indicative of the presence or absence of the wurtzites in the concretions, for they have been noted in

very narrow fractures often coating most or nearly the entire surfaces in radiating crystal growths, as at the Wittmer locality. Larger crystals up to two and one half millimeters in length have been found in the wider shrinkage cracks, for here the crystals had more space in which to grow.

Figure 1 shows the area around Shelocta, Pennsylvania, the original locality of the polymorphous wurtzites. Figure 2 is a photograph taken

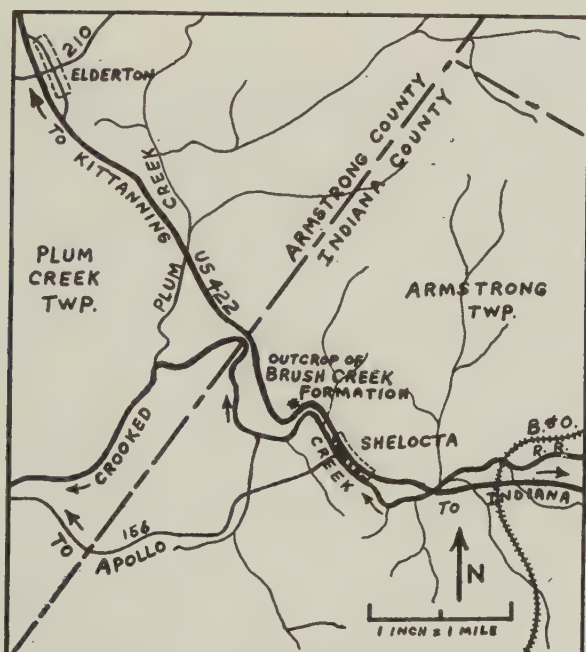


FIG. 1. Area around Shelocta, Pennsylvania.

at this locality. Figure 3 shows the geological section at the Shelocta locality. Figure 4 shows the location of all the polymorphous wurtzite localities within a fifty mile radius of Pittsburgh. Fourteen localities are shown: the one near Glouster, Athens County, Ohio, being omitted as it is some one hundred and twenty-five miles southwest of Pittsburgh.

LOCALITIES

The following is a list of all the localities so far found where the wurtzites occur. They are listed from east to west beginning with the Shelocta locality and ending with the Glouster, Ohio, locality. Their positions may be noted on the map, Figure 4.

1. One half mile northwest of Shelocta, Indiana County, Pa., along U. S. Route 422.
2. Along U. S. Route 422 about a mile southeast of Elderton, Armstrong County, Pa.
3. Railroad cut along the main line of the Pennsylvania Railroad just east of the small station of Donohoe, about four miles east of Greensburg, Westmoreland County, Pa.
4. Along the old William Penn highway about one quarter of a mile west of Murrys-ville, Westmoreland County, Pa.
5. Along a branch line of the Pennsylvania Railroad about two miles south of the William Penn highway (U. S. Route 22) at Abers Creek about eight miles east of Wilksburg, Allegheny County, Pa.



FIG. 2. Brush Creek limestone (center) near Shelocta, Pennsylvania.

6. Along a secondary road one and one half miles southwest of Murrys ville, Westmoreland County, Pa.
7. Old brickyard quarry at Valley Camp, Westmoreland County, Pa.
8. McFettridge quarry, Creighton, Allegheny County, Pa.

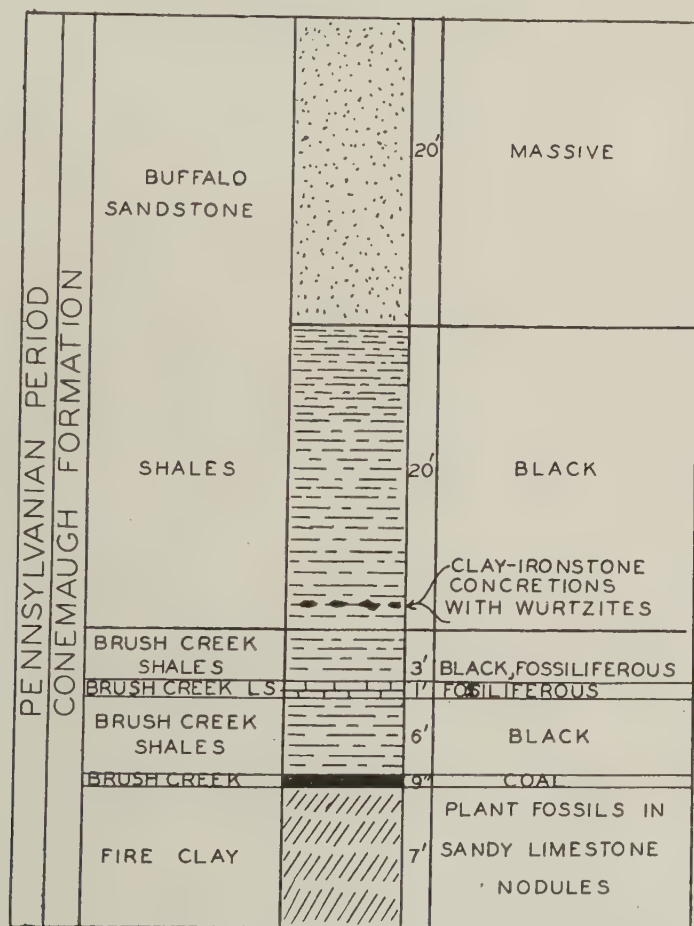


FIG. 3. Geologic section near Shelocta, Pennsylvania.

9. Glassmere Brick Company quarry at Glassmere, Allegheny County, Pa.
10. Along Route 28 about a half mile north of Springdale, Allegheny County, Pa.
11. Baltimore & Ohio Railroad cut at Wittmer, about two miles north of Etna, Allegheny County, Pa., and along Route 8.
12. West end of Sewickley bridge opposite from Sewickley, Allegheny County, Pa.
13. Junction of Flaugherty Run and the Shousetown road, one and one half miles northwest of Sewickley, Allegheny County, Pa.
14. Stanton Park along Route 7 about a mile and a half north of Steubenville, Jefferson County, Ohio.

15. Old Glouster Brick quarry, one third of a mile south of Glouster, Athens County, Ohio.

The wurtzite-bearing concretions range from a layer one foot above the Brush Creek limestone at the Sewickley and Wittmer localities to from fifteen to twenty feet above this limestone at the Creighton and Valley Camp localities. All of the strata are essentially horizontal in this area with the exception of those at the Donohoe locality which dip from east to west at about an angle of thirty-five degrees. The wurtzites are



FIG. 4. Map of wurtzite localities in Pittsburgh area.

extremely rare at some localities as at Sewickley, Creighton and Glassmere, Pennsylvania. About one in ten concretions show them at the Wittmer locality and about one in four concretions at the Donohoe locality. Peculiar expanded bulb-like terminations at the opposite end of the pyramidal terminations occur on the polymorphous wurtzites at Donohoe, Pennsylvania. A few tiny tetrahedral sphalerite crystals have been found embedded in the platy barite in the concretions at the Shelocta locality and at locality number 13 northwest of Sewickley. Chalcopyrite in tiny crystals has been noted as an associated mineral at Valley Camp. At the Wittmer locality near Etna, the Brush Creek limestone occurs about three feet above the lower bench in the railroad cut, and the Pine Creek limestone about the same distance above the upper bench separated vertically by about ninety feet of black, carbonaceous shales.

The wurtzite-bearing concretions are found in layers about one foot above both limestones in this deep cut. Here as at Donohoe, the wurtzites are usually found embedded in granular calcite which may be readily dissolved by placing the specimens in a solution of cold, dilute hydrochloric acid which makes the crystals stand out brilliantly against the clay-ironstone matrix. The dilute acid does not visibly affect the polymorphs in this short period treatment.

PARAGENESIS OF MINERALS

The paragenesis of the minerals found in the concretions appears in general to be as follows. The polymorphous wurtzites were formed first; all three types occur side by side in rudely radiated aggregates of single crystals, indicating simultaneous crystallization; then followed curved and radiating, platy sphalerite. The wurtzites have been noted at several localities, as at Valley Camp, with their steep pyramidal terminations penetrating the sphalerite plates showing that they had formed before the sphalerite. Pyrite or chalcopyrite may be next as they have been found as coatings over the previously formed minerals. Finally barite or calcite has been deposited, but not in the same concretions for all the previously formed minerals have been found embedded in one or the other at numerous localities. However the wurtzites may occur alone without any associated minerals, or with several of them, as with curved and platy sphalerite, pyrite, and calcite at Wittmer, or with curved and platy sphalerite, chalcopyrite, and barite at Shelocta.

A very interesting specimen showing tiny polymorphous wurtzites deposited between the septa of a small straight cephalopod fossil was found at the Brush Creek limestone horizon at Wittmer in June, 1947. The solutions which deposited the wurtzites no doubt filled the open siphuncle in this fossil which was about a quarter of an inch in diameter and about an inch in length. This suggests that they formed during the diagenesis of the sediment, at the time the fossil was enclosed in the concretion on the ocean floor, and not as a later introduction. The association of polymorphous wurtzites with fossils in other concretions, though not within the fossils themselves, at a number of other localities leads to the same conclusion.

Certain of the concretions at Wittmer when broken reveal round or oval-shaped siderite centers with the tiny wurtzites and other associated minerals deposited completely around them as a very thin layer. These nuclei often break free and separate like nuts from a shell when the concretions are opened. This may indicate that these minerals were formed shortly after the first deposition of clay-ironstone forming the core of the concretions, and before the final growth of these concretions was

completed. All the minerals are thought to have formed under essentially ordinary conditions of temperature and pressure at the time of formation of the concretions during sedimentation. The finding of the polymorphous wurtzites and other associated minerals in clay-ironstone concretions in clay or shale rich in carbonaceous material suggests that this type of environment was very favorable to the deposition of these minerals.

An occurrence of what appears to the writers to be tiny crystals of either wurtzite-4H or wurtzite-6H, has been noted in shrinkage cracks in clay-ironstone concretions of from four to ten inches in diameter from clay below the number six (Middle Kittaning) coal, of the Richard Immel Mine southwest of Alliance, Stark County, Ohio. This stratum is near the middle of the underlying Allegheny Formation, or some three or four hundred feet below the Brush Creek limestone horizon of the Conemaugh Formation. This occurrence was reported by W. A. Rice of Mount Union College in a paper presented May 7, 1948, before the Ohio Academy of Science meeting at the University of Toledo. It should prove very interesting should polymorphous wurtzites be found at other geological horizons within the Pennsylvanian system in other areas outside of eastern United States.

CONCLUSION

It is the writers' belief that polymorphous wurtzites will be found with careful search at most of the outcrops of the Pine Creek and Brush Creek limestone wherever the accompanying clay-ironstone concretions are encountered. A fifty square mile area in western Pennsylvania and eastern Ohio is now known where they have been noted. Since they have been found near Glouster, Ohio, some one hundred and twenty-five miles southwest of Pittsburgh, it seems likely that they will be found by careful search at localities between these two points. A diligent search should also be made of the known outcrops of these two limestones in Maryland and West Virginia.

ON THE NATURE OF THE OPAL-LIKE OUTER LAYER OF COATED DIAMONDS

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ABSTRACT

A description is given of so-called coated diamonds which consist of a clear core surrounded by an opal-like layer or coating. From a careful microscopical study of this coating, which is pure diamond, wherein minute foreign particles are embedded, a suggestion is made as to the growth pattern of the diamond. Growth is found to occur not only in waves, which include angles of 60° , but growth angles of about 80° are also observed. Attention is drawn to curious string formations of the embedded particles in the coating.

Research carried out in this laboratory has included the examination of various types of so-called coated diamonds from various sources. The literature on this subject is rather sparse and we are attempting, in the first instance, to give a clear description of the kind of stones covered by this investigation.

Almost all the coated stones, which have been examined, are of the octahedral habit and even if they are not perfect octahedrons, all the eight octahedral facets are usually present. Some stones, however, are of an irregular shape and some of them may be twins, though the percentage of this type is very low.

When looking at these stones, which may have weights of about 0.1 to 1.5 grams, they do not appear to be clear, their color being grey and dull. On opening them one can verify, however, that they contain a core or interior part which is clear diamond, though this clear part sometimes contains one or more black spots, cracks, flaws and so on.

The core is surrounded by the so-called coating. Due to its light scattering properties, this coating gives the stone its dull opal-like or cloudy appearance, thus making it quite impossible to look into the stone. As a matter of fact, the coating consists of diamond in which foreign material in the form of very tiny particles is embedded. Though we are not yet certain about the nature of this foreign matter, it is probable it is pure carbon, either in the amorphous state, or in the form of graphite. These tiny particles with a refractive index different from the index of pure diamond, act as light scattering centres. Under the binocular microscope they appear as a milky cloud in reflected light, whereas one can distinguish them as fine brown, or black-brown specks in transmitted light.

This coating shows several interesting features, which throw more-over some light on the growth of the diamond.

Usually the boundary between the coating and the clear part of the stone is very sharp. When this is the case, one can be sure that the

boundary plane is an octahedral (111) plane. The stone has grown from its center and the growth has been in layers parallel to the most densely packed "net" planes, the (111) planes. At a definite stage of the growth, precipitation of the foreign material commenced over the whole surface of an octahedral plane whilst, at the same time, the growth of the diamond crystal continued. This stage marked the start of coating formation.

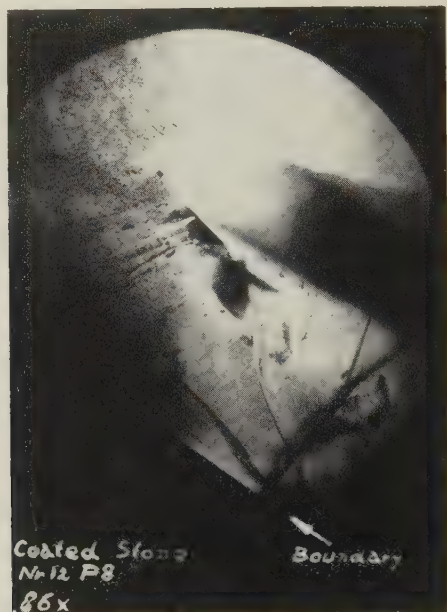


FIG. 1. Boundary between the coating and the clear part of a coated stone. Original magnification on the photograph reduced $\frac{1}{2}$ on reproduction.

The sharpness of the coating boundary is represented in Fig. 1 which is a photograph of a stone taken normally at a "window". What is meant by a "window" is illustrated in Figs. 2a and 2b. It is a facet cut on a stone and is usually orientated so that it is parallel to one side of the girdle whilst being normal to the plane of this girdle.* As is evident from Fig. 2a the window intersects normally 4 of the 8 octahedral faces and it intersects, therefore, also the four coating layers. In Fig. 1 such an intersection is shown. The border line is in most cases as sharp as a knife edge, pointing to the fact that the one layer formed is still pure diamond

* The so-called girdle of an octahedral stone is the plane of symmetry which divides the octahedron in two equal four-sided regular pyramids. It has, therefore, the form of a square and each octahedron has three girdles, the planes of which are mutually perpendicular to each other.

and that the next contains the foreign matter. The transition from the one layer to the other is less than about 0.5 micron.

Figure 2*b* gives a pair of stereoscopic pictures of an opened stone. Both the window and the cloudy coating are clearly visible and one can look through the window into the interior of the stone.

That the coating mainly consists of pure diamond and forms part of the one large single crystal occupying the whole volume of the stone is evident from *x*-ray photographs. We have not been able to find any difference on Laue photographs between the clear part of the stone and its coating. Both give rise to a Laue-photograph with well defined spots.

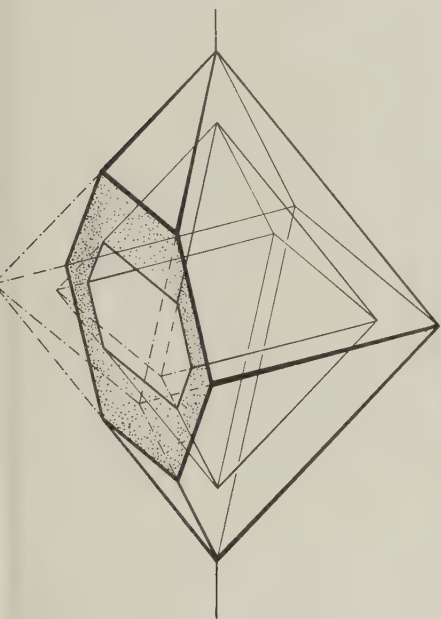


FIG. 2*a*. Drawing of a window on a coated octahedral stone. The coating is represented by dots.

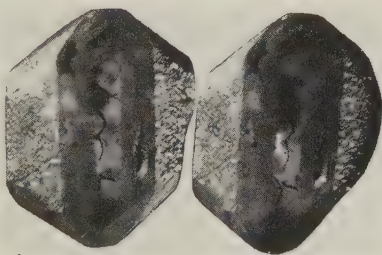


FIG. 2*b*. Stereoscopic pictures of a coated stone. The sharp boundary at the upper and lower sides can clearly be seen.

This points to the fact that the concentration of the built-in particles must be low. Otherwise, there should have been an indication of them either in the form of a pattern belonging to amorphous carbon or graphite, or to some other substance. However, when the coating is powdered and a Debye photograph taken, there is a slight indication of foreign material, for besides the ordinary diamond lines, some very faint lines with a low Bragg angle appear. We have not yet succeeded in identifying these lines.

Before going into the growth of the diamond in somewhat greater

detail, we might remark about the thickness of the coating. This may vary between about 0.005 and 0.06 inch (0.1 and 1.50 mm. respectively). There seems to be no definite relation between the weight or volume of a stone and the thickness of its coating. Most frequently the thickness is about 0.02 inch (0.5 mm.). More than once we made the observation that not only the inner boundary of the coating is clear and sharp, but the outer one also. When this is the case, the outermost layer of the stone is again pure diamond. One way to verify this is to cut away the coating of one octahedral facet —(which, by the way, is a very tedious task, the octahedral plane being the hardest with respect to abrasion),—so that one can look through the whole clear core and observe under the binocular microscope the opposite facet. After lowering the tube of the microscope, one first distinguishes the window plane, then one may focus on the inner boundary of the coating and the core, distinguishing very clearly the black particles or specks and finally one focusses on the outside of the stone, that is the opposite octahedral facet mentioned above. The growth cavities in the form of triangles which are a very common feature of nearly all these coated stones, can then be seen quite clearly, but the coating particles are no longer visible.

There are several reasons for supposing that this foreign matter built into the crystalline diamond structure is the origin of stresses and strains set up in the crystal. One can frequently observe a crack running along the boundary between the coating and the clear part of the stone. Cracks are also found in other directions, though mostly coincident with (111) planes. It is well-known that when foreign atoms are built into a crystal, they can easily give rise to stresses, especially when their crystal structure is different, or if their atomic radius has another value. This is especially valid for metals where one of the methods to harden them is alloying with different elements. The foreign atoms deform the crystal lattice and increase the lattice energy.

For crystalline structures, such as ionic crystals and homopolar bond crystals, of which diamond is an example, one may expect the crystal to become brittle and more unstable if foreign atoms are present. In this connection, it may be mentioned that this brittleness is experienced on cleaving the coatings. It is only rarely that a flat surface is the result, on the contrary, it is nearly always of an irregular appearance.

It is frequently found that the black particles of the coating are arranged on strings. Figure 3 is a reproduction of such a string formation. In this reproduction, one looks normally on to the window and this is another illustration of the sharpness of the coating boundary.

These strings lie in (111) or octahedral planes, their direction is [110] which means that they run parallel to any of the sides of the equilateral

triangle by which an octahedral face is bounded. This all points to a definite growth pattern and, if it may be assumed that the string formation is an indication of how growth occurred, the following process can be suggested. In Fig. 4 ABC is an octahedral plane, pqr s represent growth waves, and v and w give the directions of growth. These directions all belong to the form $\{110\}$. On this theory, a wave front such as s is

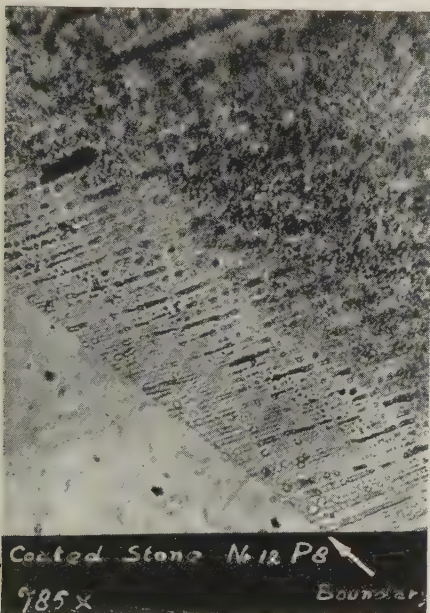


FIG. 3. String formation in a layer of coating. The boundary is indicated by an arrow. Original magnification on the photograph reduced $\frac{1}{2}$ on reproduction.

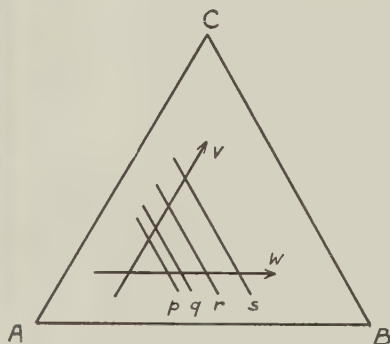


FIG. 4. Illustration of the growth of the diamond. ABC is an octahedral plane, pqr s are growth waves in the directions v and w .

parallel to a boundary line of coating, as can be seen in Fig. 3 whilst a direction of growth such as v , in Fig. 4 is parallel to the direction of the strings in Fig. 3. It is realized that this suggested theory of crystal growth of the diamond is only a theory and not a proven fact. It is, however, felt that the growth followed a definite pattern, as is made evident by this string formation, and it is a known fact that nearly all face-centered cubic crystals grow in layers parallel to octahedral planes, these being the planes of highest atomic density.

After the crystal has been built up in this way to a certain size so that flat surfaces like ABC of Fig. 4 have been formed, for some reason unknown up to now, layers were deposited containing the black particles, and it is from the arrangement of these particles in strings that this pic-

ture of the growth of the whole monocrystal is obtained. This string formation went on for some time. Then it might happen again for an unknown reason that the crystalline formation went on quite regularly, i.e. without containing foreign particles. This affords an explanation of the formation of so-called prelayers of coating. Finally, the coating proper was crystallized. Temperature and pressure conditions must have played a predominant role in this process.



FIG. 5. Coating formation in the form of "fish bones." Original magnification on photograph reduced $\frac{1}{2}$ on reproduction.



FIG. 6. Example of a regular string formation. The strings mostly include an angle of 60° . This picture shows, however, also strings which include an angle of about 80° . Original magnification on photograph reduced $\frac{1}{2}$ on reproduction.

Another example of coating formation is given in Fig. 5. Here two "fish-bone" formations can be seen. "Growth of the coating has occurred in two plane waves in each "fish-bone." The front lines of these waves can be seen to include an angle which is actually 90 degrees (angle between two different [110] directions) but is seen here as projected on a (110) plane (plane of a window), the projected angle being about 110 degrees, which is the angle between the normals to two adjacent octahedral planes. The two halves of the "fish-bone" lie in two different planes, which are not coplanar and which are also adjacent (111) planes. From Fig. 5 it can be seen that only the sides of the "fish-bone" are in focus, whereas the center line is not. This center line along the whole length of the "fish-bone," which is actually the line of intersection of the two waves, may be explained as the line at which the two waves were halted by each other. The center line indicates the direction of growth.

An optical analogy is found in the propagation of light in a doubly-refracting medium. Here the extraordinary wave is propagated in a direction which is not normal to the wave front. This curious "fish-bone" formation is very striking and occurs frequently.

In Fig. 6 the coating formation is shown under a higher magnification. Whereas the arrangement of the particles on strings is sometimes very irregular, a formation of frequent occurrence is shown here at a magnification of $795\times$. This photograph is interesting for it shows not only the growth lines including an angle of 60 degrees with each other as may be expected because the plane of these strings is a (111) plane, but it shows, moreover, growth lines including an angle of about 83 degrees with each other. This value is the average of five determinations, the extreme values being 81.0 degrees and 83.7 degrees, respectively.

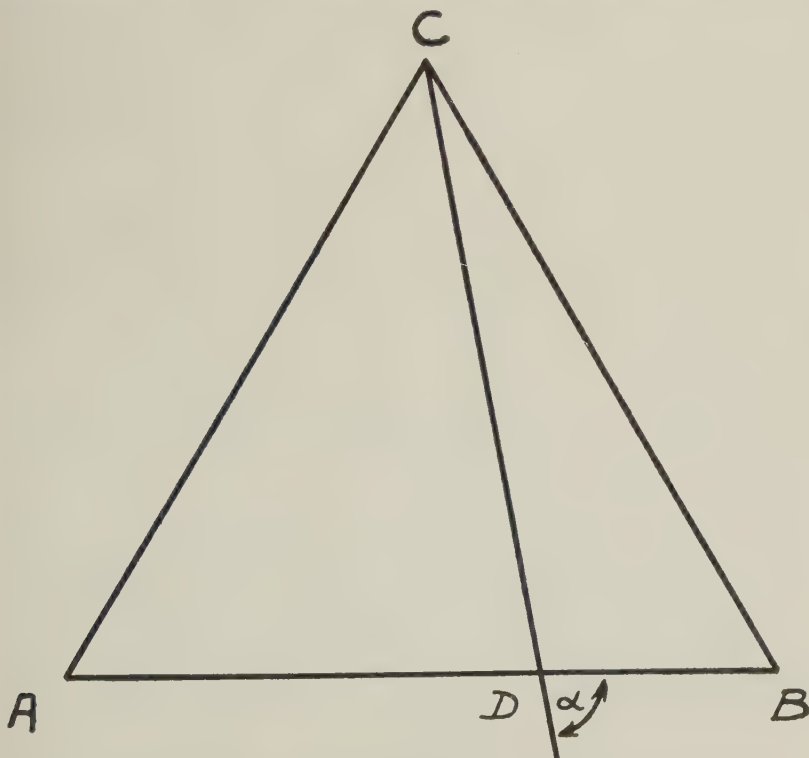


FIG. 7. The direction of the 80° strings in the octahedral plane of a coated stone is obtained by dividing AB in about three equal parts.

One may ask what crystallographic direction is coincident with these growth lines. In Fig. 7 AB , BC and CA are assumed to be growth waves. If now we divide, for example, AB in three equal parts and draw the line

CD then the angle α is about 80 degrees, to be exact $79^{\circ} 6'$. One can easily verify that the direction represented by this line is a $[123]$ direction. Assuming then that the strings represent lines along which the growth of the crystal took place, so that layers were formed parallel to octahedral planes, we must assume also that growth can take place along lines unknown up to now (the 80 degree angle). Finally we might remark that these observations support those authors who are of the opinion that the triangular pits on the surface of so many diamonds are growth cavities and not etch pits.¹ The sides of these triangles are the fronts of the growth waves which grew no further. The only remarkable point here is that many times a face contains hundreds of these triangles so that there must have been about the same number of centers from which growth started. And why did growing stop? By lack of material? Our opinion is that this cannot have been the only reason.

We have never observed growth triangles on the surface of diamonds other than equilateral ones. What we did find, however, were lines making again an angle of about 80 degrees with each other. This remarkable angle between lines of growth is, therefore, to be found not only in the interior of a crystal, but also at its surface.

In this connection, it is worth while drawing attention to an article on the cleavage properties of diamond by Ramaseshan.² This investigator established definitely the following cleavages: (111) , (221) , (110) , (322) , (331) , (211) and (332) .

One might ask if there is any relationship between the above mentioned direction $[123]$ and one or more of these cleavage planes. The only relation found is that the planes $(11\bar{1})$, $(\bar{3}3\bar{1})$ and $(1\bar{2}1)$ of the forms $\{111\}$, $\{331\}$ and $\{211\}$, respectively, belong to the zone $[123]$, though according to Ramaseshan, cleavages (221) and (110) were the most frequent after the (111) cleavage.

The author wishes to thank Dr. R. S. Young of this Laboratory for his kind interest in the progress of this investigation and Miss H. Grenville-Wells for valuable assistance in taking the x -ray pictures.

¹ Tolansky, S., and Wilcock, W. D., *Proc. Roy. Soc. London (A)* **191**, 182-194 (1947).

² Ramaseshan, S., Sec. Symp. on the Structure and Properties of Diamond, pp. 114-121. Reprinted from: *Proc. Indian Acad. of Sci.*, Bangalore (1946).

ARROJADITE, HÜHNERKOBELITE, AND GRAFTONITE*

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ABSTRACT

Headden's unnamed sodium iron manganese phosphate from the Nickel Plate pegmatite, S. Dak., has been reexamined and its identity with arrojadite from the Serra Branca pegmatite, Brazil, has been established by direct comparison. The optical properties of arrojadite from the Nickel Plate pegmatite are: $\alpha=1.664$, $\beta=1.670$, and $\gamma=1.675$; X colorless, Y pale green, Z pale yellow green, $X=b$, $Z\wedge a=18^\circ$, $2V=86^\circ$, $r<v$ strong. New optical properties of arrojadite from other localities are given. The space group is $C2/m$. (C_{2h}^3) and the unit cell has $a_0=16.60$, $b_0=10.02$, and $c_0=23.99$ Å; $a:b:c=1.656:1:2.389$, $\beta=93^\circ 37'$.

Material from Hühnerkobel, Bavaria, and from Norrö, Sweden, originally described as arrojadite is not isostructural with arrojadite from the Nickel Plate pegmatite and from the Serra Branca pegmatite, and the name hühnerkobelite is proposed for this partly oxidized material with formula $(Na_2, Ca)O \cdot 2(Fe, Mn)O \cdot P_2O_5$.

Graftonite $(Fe, Mn, Ca)_3(PO_4)_2$ is associated with arrojadite at the Nickel Plate pegmatite. A new analysis is given, together with a partial analysis of graftonite from the Rice pegmatite, and optical properties are correlated with chemical composition. Optical properties of the graftonite from the Nickel Plate pegmatite are: $\alpha=1.709$, $\beta=1.714$, $\gamma=1.736$, X colorless, Y colorless, Z pink, $X=b$, $Z\wedge c=36^\circ$, $2V=53^\circ$, biaxial+, $r<v$ strong. The space group is $P2_1/c$ (C_{2h}^6); the unit cell has $a_0=8.87$, $b_0=11.57$, and $c_0=6.17$ Å; $a:b:c=.766:1:.533$, $\beta=99^\circ 12'$. The relationship between the new cell and the Penfield orientation is discussed.

INTRODUCTION

Previous work

In the course of an investigation of phosphate minerals from the Black Hills, it was desired to know whether arrojadite was present. Published descriptions of the mineral were inadequate and appeared to describe

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different minerals. Type arrojadite from the Serra Branca pegmatite,¹ Brazil (Guimarães, 1925) was said to represent the partly oxidized form of the mineral described, but not named by Headden in 1891 (Headden, 1891) from the Nickel Plate mine, Pennington County, S. Dak. Direct comparison of the two had never been made. Quensel (1937) gave the name headdenite to the Nickel Plate mineral. The priority of the name arrojadite was pointed out by both Mason (1941, p. 132) and Guimarães (1942) though neither had access to the Nickel Plate arrojadite. Arrojadite has also been described from the Norrö pegmatite of Sweden (Ericksson, 1946) and from Hühnerkobel, Bavaria (Mason, 1942), but these minerals were called arrojadite primarily on the basis of chemical composition, and not by direct comparison with either the Brazilian or the Black Hills arrojadite. Arrojadite from various localities has quite different chemical analyses, and these should be used with caution as a means of establishing the identity of members of the species, especially when part of the iron is oxidized.

Arrojadite from the Nickel Plate mine and the Serra Branca pegmatite was obtained from the Headden collection of Harvard University and from the U. S. National Museum for direct correlation of physical properties. A sample of varulite from Skruppetorp, Sweden, and samples of arrojadite from Hühnerkobel, Bavaria, and Norrö, Sweden, were obtained from Professor Brian Mason of Indiana University.

Description of material from the Nickel Plate mine

Headden described a green phosphate from the Nickel Plate mine as being associated with beryl, spodumene, mica, and cassiterite in granite. A dark-green cleavage block about 6 inches long, from the Headden collection of Harvard University, appearing massive and homogeneous on the outside, was found to contain small books of muscovite, granular quartz, graftonite, and cassiterite in the interior.

Thin sections showed that arrojadite and cassiterite were intimately intergrown, with thin seams and some euhedral crystals of cassiterite in the cleavage planes of the arrojadite. Parallel euhedral cassiterite crystals are developed in the cleavage planes of arrojadite and in the associated quartz. The quartz and arrojadite have a curved boundary which suggests replacement of the arrojadite by quartz. Of especial interest are rows of haloes in the cassiterite. As the cassiterite itself is strongly pleochroic, any possible pleochroism of the haloes is marked. The rows of haloes in some thin sections occupy the whole width of the cassiterite

¹ As used in this paper the terms Serra Branca pegmatite, Rice pegmatite, Norrö pegmatite, Etta pegmatite, and Skruppetorp pegmatite serve only as a convenient means of reference and are not to be considered as analogous to stratigraphic names.

seam in the arrojadite cleavage planes; in other sections, the row of haloes was seen at one side or in the center of the seam. In others several rows occurred. The rows of inclusions are usually parallel to the b axis in the arrojadite. A small sample of purified cassiterite was found to be feebly radioactive, but the sample was too small to obtain quantitative results.

At some places the graftonite showed an abrupt contact with the arrojadite; at other places it was surrounded by a rim of material with relief similar to that of the graftonite, but with abnormal blue birefringence. Long, thin stringers of original arrojadite showing parallel extinction lie in the graftonite and suggest that graftonite may have replaced arrojadite along cleavage planes, though definite paragenetic relationships could not be completely determined.

In the arrojadite are occasional shreds of a deep grass green material, which has the appearance of the grass-green varulite from Skruppetorp, Sweden.

GRAFTONITE

Optical studies

The optical constants for graftonite from the Nickel Plate mine are given in Table 1. The optical orientation is given for the new axial directions as defined by single-crystal x -ray studies discussed below. In any

TABLE 1. OPTICAL CONSTANTS FOR GRAFTONITE
Nickel Plate mine, S. Dak.

Indices	Absorption	Orientation
α 1.709	X colorless	$X=b$ $2V=53^\circ$, sign +
β 1.714	Y colorless	
γ 1.736	Z pink	$Z/\wedge c=36^\circ$ $r < v$ strong

group of minerals in which isomorphous substitution occurs, it is desirable to correlate the indices of refraction with chemical composition. The chief variables in composition in graftonite are calcium, manganese, and iron. The per cent of each for all known analyzed samples is given in Table 2, together with the indices of refraction, arranged in order of increasing alpha index. It is seen that a high calcium content lowers the indices of refraction and that iron and manganese raise the indices, iron more than manganese. The indices of refraction of graftonite from Olgiasca, Lake Como, Italy, are a little low in comparison with other recorded indices. The low calcium and high ferrous iron content of this mineral indicate that it should have the highest observed indices of refraction. The very

TABLE 2. CORRELATION OF INDICES OF REFRACTION OF GRAFTONITE WITH CALCIUM, MANGANESE, AND IRON CONTENT

	1	2	3	4	5	6
α	1.695	1.700	1.705	1.708	1.709	1.709
β	1.699	1.705	1.708	1.713	1.714	1.714
γ	1.719	1.724	1.722	1.724	1.733	1.736
CaO	12.80	9.23	7.95	4.50	4.71	6.00
MnO	15.96	17.62	15.65	23.32	25.48	21.81
FeO	28.84	30.65	32.58	32.33	27.78	30.70

1. Graftonite, Rice pegmatite, North Groton, N. H., Lindberg, analyst.
2. Graftonite, Melvin Mtn., Grafton Co., N. H., S. L. Penfield, analyst (Penfield, 1900).
3. Graftonite, Valle della Madonna, Brissago, Tessin, Switzerland. (Parker, 1939).
4. Repossite, Olgiasca, Lake Como. Italy. Gallitelli, analyst (Periodico Mineral) (Grill, 1935).
5. Graftonite, Greenwood, Maine, Fahey, analyst (Glass, 1937).
6. Graftonite, Nickel Plate mine, Lindberg, analyst.

similar indices of refraction for the Greenwood, Maine, and the Nickel Plate material show the compensating effect of higher calcium and higher iron and less manganese in the Nickel Plate material. Calcium, manganese, and iron were determined on the graftonite from the Rice pegmatite especially for this study, as the indices of refraction indicated that this had a higher calcium content than any graftonite previously analyzed.

Chemical composition

The graftonite was separated from arrojadite by Clerici solution. Alteration along cleavage cracks on the graftonite produced a material of higher birefringence and lower indices of refraction. This alteration product had a lower specific gravity, and it was possible to float most of the impurity from the graftonite although a complete separation was not obtained.

The analysis of graftonite from the Nickel Plate mine (Table 3) conforms closely to the established formula $(\text{Fe,Mn,Ca})_3(\text{PO}_4)_2$. K.J. Murata examined the material spectrographically and reports, in addition, the presence of Sn, Zn, and Cu, in hundredths of 1 per cent, and the absence of Be, B, Ti, Zr, Ag, Tl, Pb, Bi, As, Sb, Ge, In, Cd, Mo, W, Cr, V, Co, Ni, La, Y, Ba, and Sr. The absence of spectrographic amounts of Tl indicates that substitution or base exchange does not occur during the treatment with Clerici solution.

TABLE 3. CHEMICAL COMPOSITION AND FORMULA OF GRAFTONITE FROM THE NICKEL PLATE PEGMATITE

	Analysis	Ratios	Oxygen equivalent	Met. equivalent	Atoms per cell
P ₂ O ₅	39.66	.2794	1.3968	.5587	7.94 (P)
Fe ₂ O ₃	none	—	—	—	—
Al ₂ O ₃	0.20	.0020	.0059	.0039	.06
FeO	30.70	.4273	.4273	.4273	6.07
MnO	21.81	.3075	.3075	.3075	4.37
MgO	0.10	.0025	.0025	.0025	.04
Li ₂ O	0.05	.0017	.0017	.0034	.05
Na ₂ O	0.28	.0045	.0045	.0090	.13
K ₂ O	none	—	—	—	—
CaO	6.00	.1070	.1070	.1070	1.52
H ₂ O	0.60				
F	0.20				
Insol.	0.16				
Total	99.76		2.2532 (O)		32.02 (O)
Less F=O	0.08				
	99.68				
Sp. gr.=	3.775				

Atoms per cell found by multiplying oxygen and metal equivalents by $1421 \times .01$.

$$\text{M. Wt.} = \frac{V (\text{in } \text{\AA}^3) \times 10^{-24} \times \text{density}}{1.6604 \times 10^{-24}} = \frac{625 \times 3.775}{1.6604} = 1421.$$

Formula $\text{R}_{12}\text{P}_8\text{O}_{32} = 4 \text{R}_3(\text{PO}_4)_2$.

Lindberg, analyst.

One-half gram graftonite from the Rice pegmatite, North Groton, N. H., was purified to determine the indices of refraction. When these were found to be the lowest observed indices, a partial analysis was made on the remaining portion of the sample: 15.96 per cent MnO, 28.84 per cent FeO, 12.80 per cent CaO, 41.65 per cent P₂O₅; total 99.25 per cent.

Graftonite is completely soluble in dilute HCl, HNO₃, and H₂SO₄, except that of high calcium content, which precipitates gypsum in the presence of sulfuric acid.

The molecular weight of Nickel Plate graftonite is 353.

X-ray studies

Two sets of rotation and Weissenberg photographs were taken on a small cleavage fragment so oriented that (1) the X-optical direction = *b* was the axis of rotation, and (2) {010} and {100} were in a zone parallel

to the axis of rotation $=c$, $Z \wedge c = 36^\circ$ in acute angle (new cell). Two cleavages were observed: $\{010\}$ good and $\{100\}$ fair. The unit-cell dimensions are a_0 8.87, b_0 11.57, and c_0 6.17 Å, with $\beta = 99^\circ 12' \pm 15'$. The volume of the unit cell is 625 Å³. Four molecules per unit cell were computed from the observed gravity 3.775. Examinations of projections of Weissenberg photographs showed (hkl) all orders present; $(h0l)$ present when $l = 2n$; and $(0k0)$ present when $k = 2n$. The space group is $P2_1/c(C^{5}_{2h})$.

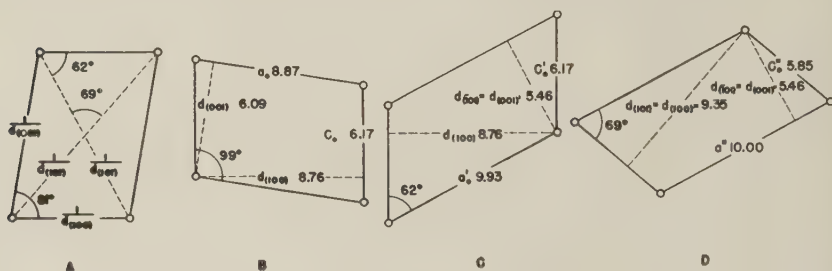


FIG. 1. ORIENTATION OF GRAFTONITE

The directions of a and c were chosen, according to x -ray convention, so as to give the unit cell with the most orthogonal β . The ratio $a:b:c = 0.766:1:0.533$, $\beta = 99^\circ 12'$. This is a different orientation of the unit cell from that chosen by Penfield (Penfield, 1900) $a:b:c = .886:1:.582$, $\beta = 66^\circ$. Penfield considered his measurements made with a contact goniometer on large crystals intergrown with triphylite as approximate only: "The fundamental measurements are not very reliable, and the axial ratios therefore can only be approximately correct." The zero layer about the b axis of rotation was examined to see what directions made angles approximately at 66° . Two sets of directions, one with a β^* of 62° and the other with a β^* of 69° were found. Figure 1 shows the reciprocal cell (A) and the corresponding direct lattice cell (B), patterned after Buerger (1942). If the direction for c remains unchanged, and the short diagonal of the unit cell becomes a , (C) shows a possible orientation corresponding to that of Penfield. If the directions of both a and c are changed to become the long and short diagonals of the unit cell projection, a second orientation (D) is also possible. Reference to Fig. 1 shows that cell (b) is most nearly orthogonal. Table 4 shows the relationship between d values calculated from measurements made from the zero level of Weissenberg films around the b axis of rotation, cell edges, axial ratios, and β .

The x -ray powder-diffraction spacing data from graftonite from the Nickel Plate mine are given in Table 5. For comparison, spacing data are given for graftonite from Greenwood, Me. (U. S. Nat. Museum No. 103008; analyzed by J. J. Fahey (Glass, 1937), and graftonite from the Rice pegmatite, North Groton, N. H.

TABLE 4. ORIENTATION OF GRAFTONITE

	New orientation \AA	Possible old orientation		Penfield orientation
		1st choice \AA	2nd choice \AA	
$d_{(100)}$	8.76	8.76	9.35	
$d_{(001)}$	6.09	5.46	5.46	
β^*	$80^\circ 48'$	62°	69°	66°
a_0	8.87	9.93	10.00	
b_0	11.57	11.57	11.57	
c_0	6.17	6.17	5.85	
β	$99^\circ 12'$	118°	111°	114°
$a:b:c$	0.766:1:0.533	0.858:1:0.533	0.866:1:0.506	0.886:1:0.582

TABLE 5. X-RAY POWDER SPACING DATA FOR GRAFTONITE—
IRON RADIATION, MANGANESE FILTER (\AA)

Nickel Plate, S. D.		Rice Pegmatite, N. H.		Greenwood, Me.	
I	d	I	d	I	d
2	4.31	3	4.31	3	4.30
1	3.61				
9	3.50	9	3.52	9	3.50
1	3.36				
1	3.17	1	3.14		
1	3.08				
4	3.02	4	3.01	4	3.01
4	2.956	4	2.966	3	2.953
2	2.902			1	2.906
10	2.860	10 (Broad)	2.899	10	2.867
		4	2.860		
1	2.810				
7	2.715	8	2.706	7	2.715
1	2.550				
1	2.510	1	2.510	1	2.52
		1	2.464		
3	2.412	3	2.405	2	2.410
2	2.377			1	2.377
1	2.311	1	2.323	1	2.311
1	2.271	1	2.271	1	2.276
1	2.230			1	2.226
2	2.133	1	2.126		
		1	2.082		
2	2.067	1	2.052	1	2.058
2	2.042			1	2.037
1	1.970	1	1.972		

TABLE 5—(continued)

Nickel Plate, S. D.		Rice Pegmatite, N. H.		Greenwood, Me.	
<i>I</i>	<i>d</i>	<i>I</i>	<i>d</i>	<i>I</i>	<i>d</i>
2	1.927	2	1.927	2	1.927
1	1.892	1	1.886	1	1.887
1	1.875			1	1.875
1	1.836	2	1.850	2	1.831
1	1.802			1	1.806
1	1.788	1	1.774	1	1.778
2	1.757	2	1.769	1	1.760
1	1.733	1	1.735	1	1.728
1	1.711	1	1.715	1	1.717
2	1.687	1	1.693	1	1.687
1	1.658	1	1.659	1	1.657
2	1.617	1	1.619	2	1.619
1	1.600	1	1.600	1	1.600
1	1.577	1	1.584		
1	1.549	1	1.539		
1	1.543				
2	1.516	1	1.524	2	1.501
2	1.495	2	1.495	2	1.481
1	1.453			1	1.455
1	1.424			1	1.436
1	1.411	1	1.414	1	1.414
1	1.386			1	1.397
1	1.371	1	1.366		
1	1.351	1	1.351	1	1.356
1	1.337			1	1.327
2	1.315	2	1.319		
1	1.306	2	1.314	1	1.301
1	1.287				
2	1.251	1	1.248		
1	1.231	1	1.213	1	1.236
1	1.205	1	1.205		
1	1.200				
1	1.160	1	1.170		
1	1.156	1	1.157		
1	1.103	1	1.102		
1	1.083				
1	1.061				
1	1.057				
1	1.047				
1	1.012				
1	0.9882				
1	0.9864				
1	0.9735				

ARROJADITE

Optical properties

The optical properties and the chemical composition of arrojadite from the Nickel Plate mine, S. Dak., are listed in Table 6, together with newly determined indices of refraction of arrojadite from the Serra Branca pegmatite, Brazil, and from the Etta pegmatite, S. Dak. In addition, the published optical properties and chemical composition of varulite and oxidized related minerals are given. The indices of refraction of the Nickel Plate arrojadite are found to be almost identical with those of arrojadite from the Etta mine, and slightly higher than the newly determined indices of arrojadite from the Serra Branca pegmatite (U.S.N.M. 96111). Guimarães (1942) obtained lower indices, but his low indices are a correction of his earlier reported higher indices ($\gamma=1.70$; $\gamma-\alpha=.007$) (Guimarães 1925). Guimarães' optical orientation is incorrect, possibly due to misidentification of directions and planes which are here identified by taking single-crystal x -ray pictures about certain directions previously oriented optically. In a monoclinic mineral, the optic plane cannot coincide with $\{110\}$. The new optical orientation is $X=b$; $Z \wedge a=18^\circ$. The best cleavage is $\{001\}$; $\{201\}$ is a fairly well developed cleavage; the angle between the two is 68° . Z lies in the acute angle.

In any group of minerals in which substitution occurs, it is desirable to correlate chemical composition with indices of refraction. Despite wide differences in chemical composition between the arrojadite from the Nickel Plate and Serra Branca mines, the indices of refraction differ by no more than 0.003, and it is very likely that an analysis made upon a pure sample of the latter might show less oxidation of the iron. Samples of so-called arrojadite from Europe have much higher indices of refraction, and if they are considered in a separate series, which excludes the Serra Branca arrojadite, they show increasing indices with increasing ferric iron, decreasing phosphate, and decreasing manganese. The substitution of large amounts of calcium for sodium in the Hühnerkobel, Bavaria, arrojadite probably also contributes to its higher indices. Increasing birefringence may accompany this change in indices, but enough optic data for the series are not yet known.

The absorption is so weak in the Nickel Plate and Serra Branca arrojadite, that pleochroism can be observed only in thick pieces. In the European material, with the exception of the type varulite from Varuträsk, the absorption is very strong grass green. The arrojadite from Hühnerkobel and Norrö occurs in dense, very fine grained masses, whereas the arrojadite from the Nickel Plate and Serra Branca pegmatites is coarsely crystalline, cleavage pieces 6 inches in length being known.

TABLE 6. OPTICAL PROPERTIES AND CHEMICAL COMPOSITION OF ARROJADITE AND OXIDIZED RELATED MINERALS

Indices	1	2	3	4	5	6	7	8
α	1.649	1.662	1.665	1.664	1.708	1.718	1.720	1.754
β	1.654	1.668	1.670	1.670				
γ	1.657	1.672	1.675	1.675	1.722	1.731	1.732	>1.770 probably 1.785
<i>Absorption</i>								
X	weak colorless	weak	weak	weak colorless	strong	strong green yellow	colorless	strong gray green
Y	colorless			pale green				
Z	pale green			pale yellow green		grass green		dark green
<i>Orientation</i>								
X	$c = 34^\circ$			$= b$				
Z plane	(110)			(001) = 18°				
2V	82°	80°	90°	86°	Large		70°	
<i>Sign</i>	—	—	(\pm)	—	+			
<i>Dispersion</i>				$r < v$			$r > v$	
<i>X-ray</i>		true arrojadite	true arrojadite	true arrojadite	different	different		different
<i>Composition</i>								
P ₂ O ₅	34.32			40.00	44.60	41.93	42.80	39.40
Fe ₂ O ₃	12.39			none	6.44	14.45	8.35	26.49
Al ₂ O ₃				2.66			0.36	
FeO	19.84			28.22	12.01	8.15	7.52	7.09
MnO	12.33			15.78	21.06	9.79	25.30	6.44
CaO	5.69			2.46	3.60	1.35	4.86	9.70
MgO	1.85			1.04	0.09	2.55	0.00	0.68
Na ₂ O	4.67			6.40	9.72	9.67	7.12	3.73
K ₂ O	1.45			1.74	trace	0.06	0.12	0.05
Li ₂ O	trace			0.09		0.25	0.88	0.36
H ₂ O+	4.96			0.91	1.52	0.85	0.75	4.49
H ₂ O-	0.44			none	0.14	0.20	0.14	0.24
F				0.80	0.08	0.00	0.06	
Insol.	0.66			0.11	0.44	0.50	1.80	1.88
SnO ₂	1.52							
Total	100.12			99.87	99.70	99.75	100.06	100.55
Sp. gr.				3.553		3.55		3.45

1. Arrojadite, Serra Branca, Brazil. (Guimarães, 1942.)
2. Arrojadite, Serra Branca, Brazil. U.S.N.M. No. 96111, optics by Lindberg.
3. Arrojadite, Etta pegmatite, South Dakota. Harvard No. 523, optics by Lindberg.
4. Arrojadite, Nickel Plate mine, South Dakota. Harvard No. 543, optics and analyses by Lindberg.
5. Varulite, Skruppetorp, Sweden. (Mason, 1940.)
6. Arrojadite, Norrö pegmatite, Sweden. (Eriksson, 1936.)
7. Varulite, Varuträsk, Sweden. Type material. (Quensel, 1937.)
8. Arrojadite, Hühnerkobel, Bavaria. (Mason, 1942.)

Chemical composition

A piece an inch wide was cleaved from the end of a large block of the Nickel Plate sample, and a fresh unoxidized sample of arrojadite was prepared from the interior. Impurities were removed by the use of methylene iodide and Clerici solution; the sample was washed until the washings gave no test for thallium. A small portion of the sample was then dissolved and gave no test for thallium. The sample so obtained for chemical analysis is at least 99.5 per cent pure.

The new chemical analyses and ratios are given in Table 7 together with Headden's old analysis. Although the author analyzed material from the Headden collection of Harvard University, certain discrepancies were found to exist between the two analyses. The greater purity of

TABLE 7. CHEMICAL ANALYSIS AND FORMULA OF ARROJADITE,
NICKEL PLATE MINE, S. DAK.

	1	2	Ratios	Calculations of Analysis		
				Oxygen equivalent	Met. equivalent	Atoms per cell
P ₂ O ₅	38.64	40.00	.2818	1.4090	.5636	48.02
Fe ₂ O ₃	none	none				
Al ₂ O ₃		2.66	.0261	.0783	.0522	4.45
FeO	25.05	28.22	.3928	.3928	.3928	33.47
MnO	15.54	15.78	.2226	.2226	.2226	18.97
MgO	1.50	1.04	.0258	.0258	.0258	2.20
CaO	5.53	2.46	.0439	.0439	.0439	3.74
Li ₂ O	0.28	0.09	.0030	.0030	.0060	.51
Na ₂ O	7.46	6.40	.1032	.1032	.2065	17.60
K ₂ O	2.00	1.74	.0185	.0185	.0369	3.14
H ₂ O	0.73	0.91	.0505	.0505	.1010	8.61
F	0.69	0.80	.0421	— .0211		3.59
Insol.	2.47	0.11				
Total	99.89	100.21	Total O	2.3265		198.24 (O)
Less O = F	.23	0.34				
	99.66	99.87				
Sp. gr.		3.553				

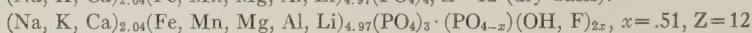
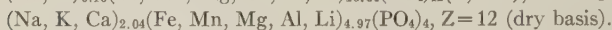
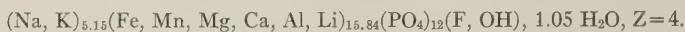
Atoms per cell found by multiplying oxygen and metal equivalents by $8521 \times .01$.

$$\text{M Wt.} = \frac{\text{Vol.} \times \text{density}}{1.6604} = \frac{3982 \times 3.553}{1.6604} = 8521.$$

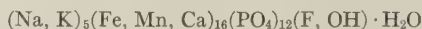
1. Headden, analyst.
2. Lindberg, analyst.

the author's sample is indicated by the smaller amount of insoluble material (2.47 per cent in Headden's analysis, 0.11 per cent in the new analysis). In addition to insoluble impurity it is very likely that small amounts of graffonite or other soluble impurities existed in his sample.

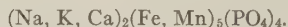
The number of atoms of each kind per unit cell is given in the last column of Table 7. In deriving a formula for arrojadite, it must be considered (1) whether calcium occupies a position equivalent to other divalent ions, or whether on the basis of ionic radius it should be grouped with sodium and potassium; and (2) whether water present occurs in the mineral as water of crystallization or as hydroxyl. Previous authors (Mason 1941 and Headden 1891) have calculated the formula of arrojadite on a dry basis. Formulas based on the above considerations are:



The ratios were obtained by grouping the number of atoms per cell (last column Table 7), and by dividing through by Z , the number of molecules per unit cell. The space group imposes the requirement that Z be an even number of molecules. The simplified formula for (1) can be written



and for (2) and (3)



Arrojadite is completely soluble in dilute HCl , HNO_3 , and H_2SO_4 .

X-ray studies

Two sets of rotation and Weissenberg photographs were taken on a small cleavage fragment so mounted that (1) the x -optical direction ($=b$) was the axis of rotation, and (2) $\{001\}$ and $\{010\}$ were in a zone parallel to the axis of rotation $= [100]$. The a and c directions were so chosen as to make a c -centered cell with $\beta = 93^\circ 37' \pm 15'$. The best cleavage is $\{001\}$; $\{201\}$ is a fairly well developed cleavage; the angle between the two cleavages is 68° . The unit-cell dimensions are a_0 16.60, b_0 10.02, and c_0 23.99 Å; $a:b:c = 1.656:1:2.389$. The volume of the unit cell is 3982 Å³. Examinations of projections of Weissenberg photographs show (hkl) present when $h+k$ even, ($h0l$) present when h even and ($0k0$) present when k even. Three possible space groups are C 2/ m , C_2 and C_m (C_{3h}^3 , C_2^3 , C_s^3). Since arrojadite is apparently not piezoelectric,*

* C. Frondel, private communication.

$C 2/m$ is favored. Additional absences are: ($h00$) with h not divisible by 4, ($00l$), l not divisible by 2, and ($h0l$), $h/2+l$ odd.

In Table 8 are given the x -ray powder spacing data for arrojadite and related minerals. The arrojadite from the Nickel Plate mine, Pennington County, S. Dak. represents the pure analyzed sample. It has the same powder pattern as arrojadite from the Serra Branca pegmatite, Brazil (U.S.N.M. No. 96111), and the identity of the two is thus established by direct comparison of the two minerals. Also included for comparison are new x -ray data for so-called arrojadite from the Norrö pegmatite, Sweden, and from Hühnerkobel, Bavaria, and varulite from Skruppetorp, Sweden, and from Varuträsk, Sweden. Microscopic examination shows that the material from the Norrö pegmatite contained a small amount (under 10 per cent) triphylite. The sample from Hühnerkobel was part of the analyzed sample (Mason, 1942) and contained a small amount of alluaudite. The varulite from Varuträsk, supplied through the courtesy of Professor Quensel corresponds to the yellow green phosphate described by him as representing beginning alteration to alluaudite (Quensel, 1937, p. 94). No mineral, colorless under the microscope, with indices α 1.720 and γ 1.732 was noted. These latter four are remarkably similar, though some differences, in part due to the purity of the samples, occur. All four are more alike than they are similar to true arrojadite. True arrojadite shows many lines, some relatively intense, that are not present in the material from Varuträsk, Skruppetorp, Norrö, and Hühnerkobel, as is illustrated in Fig. 2. It is here proposed to consider arrojadite from the Serra Branca pegmatite and the Nickel Plate mine as isostructural, and to consider the minerals from the Norrö pegmatite and from Hühnerkobel as isostructural with the varulite from Skruppetorp, and Varuträsk, but to consider the two groups as no longer isostructural with each other. Truly they are separate but related minerals. Mason (1941) in computing formulas for varulite and arrojadite, recognized the partial oxidation of his samples as being different from the Nickel Plate analysis, but did not have type material, and could not foresee the small differences in pattern between the two types.

In addition, the x -ray spacing data for a triplite sample, Serra Branca pegmatite, obtained through the courtesy of the National Museum, Departamento Nacional da Produção Mineral, Rio de Janeiro, Brazil, is given. Two other samples from this museum (Nos. 3618 and 3619) were also triplite. These samples are probably similar to the source material for the paper by Fornaseri (1943), as Fornaseri concludes that data obtained from rotation and Weissenberg photographs prove the identity of arrojadite and triplite, and publishes unit-cell dimensions and an analysis not of arrojadite but of triplite. His values for the unit cell are: a_0 5.99, b_0

TABLE 8. X-RAY POWDER SPACING DATA
Iron radiation, manganese filter (Å)

Arrojadite Serra Branca Pegmatite, Brazil; U.S.N.M. 96111	Arrojadite Nickel Plate mine, Pennington County, South Dakota; Harvard 543 Pure	Varulite Skrumpe- torp, Sweden	Varulite Varuträsk, Sweden	So-called Arrojadite, Norrö pegmatite, Sweden; 90% pure, 10% tri- phyllite	So-called Arrojadite, Hühnerkobel Bavaria; Analyzed sample; contains alluaudite	Triplite, Serra Branca Pegmatite, Brazil	Natro- philite, Branch- ville, Con- necticut; Harvard 95263
<i>I</i> <i>d</i>	<i>I</i> <i>d</i>	<i>I</i> <i>d</i>	<i>I</i> <i>d</i>	<i>I</i> <i>d</i>	<i>I</i> <i>d</i>	<i>I</i> <i>d</i>	<i>I</i> <i>d</i>
1 12.08	2 12.14				1 16.06		
3 7.65	3 7.62		1 8.30		6 8.76		
1 7.12	1 7.12				1 8.30		
1 6.51	2 6.52	5 6.33	3 6.35	5 6.26	5 6.30		
4 5.93	4 5.93				1 5.58		
4 5.55	4 5.54	2 5.47	3 5.46	3 5.44	2 5.44		1 5.25
4 5.02	3 5.01			1 5.27			
3 4.58	3 4.58		1 4.30				5 4.51
1 4.22	1 4.23		1 4.14	2 4.28	1 4.36	1 4.27	6 4.04
1 3.84	1 3.84			1 3.92		4 3.62	1 3.92
3 3.42	3 3.42	4 3.50	4 3.498	5 3.48	3 3.49	2 3.41	4 3.66
1 3.32	1 3.33				3 3.33	6 3.23	
					2 3.21		
6 3.22	6 3.22	3 3.11	3 3.12	3 3.12	2 3.08	8 3.03	
1 3.13	2 3.13	1 3.08	1 3.08	1 3.01	1 3.02	10 2.86	
10 3.04	10 3.04	1 2.95	1 2.96	1 2.942	1 2.913	1 2.83	8 3.15
4 2.487	4 2.85	2 2.90	3 2.909	2 2.880	1 2.853	1 2.73	10 2.867
4 2.766	4 2.77	1 2.79	2 2.792	1 2.766	1 2.772	1 2.69	
8 2.712	8 2.72	10 2.73	10 2.737	10 2.712	10 2.718	2 2.60	
1 2.680	1 2.68	1 2.62	1 2.635	1 2.672		1 2.53	
1 2.594	1 2.59				1 2.580	1 2.471	8 2.608
3 2.554	3 2.56	4 2.547	4 2.556	6 2.531	3 2.526	1 2.377	8 2.586
2 2.518	2 2.518	1 2.508	1 2.506	1 2.500		1 2.316	1 2.494
2 2.421	2 2.424		1 2.432			1 2.290	3 2.420
1 2.385	1 2.391		1 2.359			2 2.218	
	1 2.309					1 2.179	
	1 2.305		1 2.212			2 2.148	3 2.377
1 2.214	1 2.226	1 2.145	2 2.152	1 2.137	1 2.175	3 2.108	3 2.318
1 2.192	1 2.202	2 2.117	2 2.215	1 2.101	1 2.148	2 2.012	3 2.262
1 2.115	1 2.156	1 2.074	2 2.070	1 2.060	1 2.087	1 1.975	
1 1.976	1 1.989	1 1.993	1 1.999	1 1.982	1 2.060	1 1.917	
1 1.955	1 1.964	1 1.961	1 1.967	1 1.952	1 1.949	1 1.833	1 1.911
2 1.915	2 1.92	1 1.935	1 1.937	1 1.859		1 1.825	1 1.851
1 1.875	1 1.888	1 1.831	1 1.832	1 1.815		1 1.815	5 1.836
1 1.857	1 1.865	1 1.795	1 1.796		1 1.824	1 1.788	1 1.818
3 1.757	3 1.767	1 1.757	2 1.750	1 1.750	1 1.795	1 1.765	3 1.744
1 1.715	1 1.719	1 1.738	1 1.742		1 1.739	1 1.744	
1 1.687	1 1.687	1 1.687		1 1.671		1 1.672	5 1.695
	3 1.664	1 1.656	1 1.666	1 1.652		2 1.638	
1 1.650	2 1.654					2 1.627	3 1.648
2 1.610	2 1.616	1 1.606	1 1.613	1 1.623	1 1.618	1 1.600	1 1.606
1 1.590	1 1.597			1 1.600		2 1.587	2 1.587
1 1.572	1 1.58	1 1.575	1 1.577			2 1.580	
1 1.566	1 1.562			1 1.565	1 1.568	1 1.565	
1 1.553	1 1.539	1 1.544	1 1.548	1 1.534		1 1.524	2 1.535
	1 1.529	1 1.524	1 1.528			1 1.520	
1 1.501	1 1.506						
1 1.485	2 1.492		1 1.499				
1 1.478	1 1.478		1 1.484				
	1 1.463	1 1.459	1 1.462			1 1.471	1 1.460
1 1.456	1 1.448					3 1.453	1 1.443
	1 1.432	1 1.435	1 1.437			1 1.435	1 1.419
1 1.424	1 1.421			1 1.419		1 1.414	1 1.401
1 1.389	1 1.388		1 1.401			1 1.394	1 1.387
	1 1.378	1 1.368	1 1.375				1 1.375
1 1.353	1 1.360						
	1 1.338	1 1.340	1 1.341				1 1.349
	1 1.330		1 1.315			1 1.330	1 1.336
	1 1.255		1 1.299			1 1.242	1 1.308
1 1.240	1 1.243					1 1.227	
	1 1.205					1 1.196	1 1.206
	1 1.195						1 1.158
	1 1.183						1 1.135
	1 1.161	1 1.147		1 1.139		1 1.090	1 1.090
	1 1.108					1 1.085	2 1.072
	1 1.094						1 1.065
	1 1.052						1 1.038
	1 1.040					1 1.041	3 1.027
	1 1.013					1 1.017	1 .981

6.80, and c_0 5.01 Å, $\beta = 73^\circ 42'$. X-ray powder spacing data of natrophilite (Harvard No. 95263) show a more complete structural difference from arrojadite than is suggested by statements by Brian Mason that natrophilite is distinguished from varulite and arrojadite by the presence of calcium in the latter:

"The essential feature which distinguishes varulite and arrojadite from natrophilite is the presence of calcium. The question thus arises as to what part this calcium plays in the structure of varulite and arrojadite.

"Thus three names are current for the iron-rich members of the arrojadite varulite group—soda triphylite, arrojadite, and headdenite. Application of the law of priority

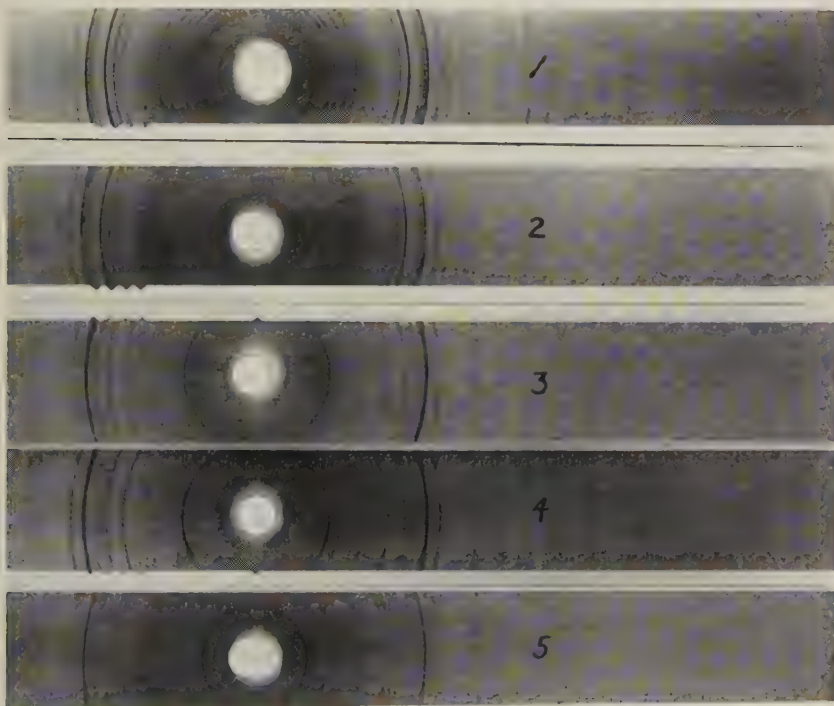


FIG. 2. X-ray powder diffraction photographs of arrojadite and related minerals. 1. Arrojadite from the Nickel Plate mine, South Dakota. Pure. 2. Arrojadite from the Serra Branca pegmatite, Brazil. Pure. 3. Varulite from the Skruppetorp pegmatite, Sweden. Pure. 4. Hühnerkobelite (previously called arrojadite) from the Norro pegmatite, Sweden. Contains triphylite as impurity. 5. Hühnerkobelite (previously called arrojadite) from Hühnerkobel, Bavaria. Contains alluaudite as impurity.

would assign the name soda triphylite to these iron-rich minerals. That is all that can be said in favor of Ziegler's name. In other respects it is thoroughly a bad name. The name soda-triphylite, if it suggests anything, suggests a mineral in which the lithium of triphylite has been replaced by sodium; this mineral is already known and has been named natrophilite. Headden's phosphate differs from natrophilite in its calcium content, and this calcium is its distinguishing feature."

Definition of arrojadite

Arrojadite from the Nickel Plate pegmatite and from the Serra Branca pegmatite are isostructural. The material from Hühnerkobel, Bavaria, and from Norrö, Sweden, are isostructural with varulite from Skrumpetorp. The relationship of the varulite from Skrumpetorp and type varulite from Varuträsk is not clear. Quensel's (1937) original description of varulite states:

"Most grains are intimately interwoven with a yellowish green phosphate, without doubt alluaudite, representing beginning alteration to that mineral."

Samples of varulite supplied through the courtesy of Professor Quensel appear to be the yellowish green phosphate and are isostructural with the Skrumpetorp varulite.

The varulite from Skrumpetorp as well as the arrojadite from Hühnerkobel and Norrö are fine-grained green minerals which could correspond either to the varulite or alluaudite of Quensel's original description of varulite. However, Mason (1942) describes arrojadite from Hühnerkobel to be associated with alluaudite; therefore, if his mineral is not arrojadite it also is not alluaudite. His description of the two minerals follows:

"The arrojadite was first identified by x-ray powder photographs, which showed its structural identity with the varulite from Varuträsk; the predominantly iron-rich nature of the phosphate minerals at Hühnerkobel suggested that in this specimen the mineral was the iron-rich member of the isomorphous series, arrojadite, rather than the manganese-rich member, varulite. This was later confirmed by chemical analysis.

"Thin sections showed that, besides being associated with triphylite and eosphorite the arrojadite was intimately mixed with about 10–20% of alluaudite, evidently the oxidation product of the arrojadite; one section also showed a small amount of a mineral wine-red in section, almost certainly heterosite formed by the further oxidation of the alluaudite. A small amount of a black mineral, probably a mixture of iron and manganese oxides, occurred along the cracks in the other minerals. The arrojadite was fine-grained, pleochroic in shades of green, and showed no cleavage; the alluaudite was also fine-grained and pleochroic in yellow and brown. As far as the paragenesis of the minerals can be read from the sections of one small specimen, it appears to be as follows: the arrojadite has been formed from and replaces triphylite, the triphylite being a crystallographic unit, the scattered remnants of which extinguish uniformly over large areas. The first stage in the replacement appears to be the changing of the triphylite to a clear mineral with a slightly yellow tinge, faintly pleochroic and with birefringence about 0.01. There is very little of this mineral in the sections as it is apparently rapidly changed to the green arrojadite, and the arrojadite is in turn further changed to the yellow alluaudite. The black opaque mineral is latest of all, and is probably not derived from the arrojadite and alluaudite of the section, but is rather a late infiltration. None of the sections showed the relationship between eosphorite and the surrounding arrojadite, but the general features of the specimen suggest that the eosphorite is later. The refractive indices of the arrojadite are: $\alpha = 1.754 \pm 0.003$, $\gamma > 1.770$, probably about 1.785. The pleochroism is strong, X = pale gray-green, Z = dark green. All the indices of the alluaudite are greater than 1.770; the pleochroism is X = dark yellow, Y = yellow brown, Z = brown."

Mason's description of arrojadite is that of a mineral altered from triphylite. The arrojadite from the Norrö pegmatite is also a mineral associated with triphylite. The arrojadite from the Nickel Plate is a primary unaltered mineral with a distinctive x-ray pattern. It is here proposed to give the name hühnerkobelite to the material from Hühnerkobel and from Norrö, not isostructural with true arrojadite.

The varulite from Skrumpetorp is isostructural with hühnerkobelite and with the yellowish green material of the varulite samples from Varuträsk. These may be considered the manganese equivalent of hühnerkobelite. A manganese equivalent to arrojadite has not yet been verified, but the possibility is suggested that the colorless mineral of Quensel's original description of varulite may be equivalent to arrojadite.

SUMMARY

Optical, chemical, and x-ray studies were made upon graftonite and arrojadite from the Nickel Plate mine, Pennington County, South Dakota. The arrojadite from the Nickel Plate pegmatite was found to be isostructural and identical with arrojadite from the Serra Branca pegmatite. Arrojadite from the Nickel Plate mine was found to be structurally different from arrojadite from Hühnerkobel, Bavaria, and Norrö, Sweden, and are designated hühnerkobelite to differentiate them from true arrojadite.

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The author is indebted to Clifford Frondel of Harvard University for making available the arrojadite and graftonite from the Nickel Plate mine and the graftonite from the Rice pegmatite, and for his many valuable suggestions in the x-ray studies. Brian Mason, of Indiana University, kindly lent the author varulite and arrojadite samples from Skrumpetorp, Sweden, the Norrö pegmatite, Sweden, and from Hühnerkobel, Bavaria. William Foshag of the U. S. National Museum lent the arrojadite from the Serra Branca pegmatite. Percy Quensel of the University of Stockholm contributed specimens of varulite from the Varuträsk pegmatite. K. J. Murata, of the U. S. Geological Survey, spectrographed the Nickel Plate graftonite. Joseph M. Axelrod and Joseph J. Fahey, of the U. S. Geological Survey, made many helpful suggestions.

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THERMAL STUDY OF POTASH-SODA FELDSPARS*

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ABSTRACT

The temperatures required to homogenize lamellar feldspars from various types of rock have been determined. The results obtained indicate that the temperatures which are necessary to effect homogenization of the specimens do not in all cases represent the minimum temperature at which the feldspars crystallized. Many of the finely lamellar feldspars may have formed by simultaneous crystallization of soda feldspar and potash feldspar rather than by the exsolution of an initially homogeneous feldspar.

INTRODUCTION

In the course of investigating the origin of a number of plutonic rocks, the writer undertook a thermal study of several lamellar feldspars¹ from metamorphic and igneous rocks in the hope of obtaining information on the temperature of formation of different rock-types. It has generally been accepted (8) that finely lamellar feldspar of the "string" type is of exsolution origin and that the temperature of crystallization of the original homogeneous feldspar is equal to, or is above, the temperature at which the heated lamellar feldspar becomes homogeneous. However, the results of the present study indicate that these assumptions are not valid for all finely lamellar feldspars. Attempts to explain the thermal behavior of the treated specimens have been only partially successful because of insufficient data, especially on the structure of the feldspars.

EXPERIMENTAL METHOD

The amount of solution of the soda feldspar lamellae at a given temperature was determined by noting the decrease in the size of the lamellae after the specimens had been heated. In the schillerized specimens the decrease in the intensity of the schiller was noted. Spencer's (8) method of determining the amount of solution by the decrease in refractive indices proved to be unreliable.

The specimens were crushed to minus 60 mesh to facilitate ready examination under the microscope by the immersion method. All of the optical properties given for the microperthites refer to the aggregate effect of the two feldspar components. The refractive indices were measured to $\pm .0005$ by the dispersion method. In order to observe the changes in

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¹ The term lamellar feldspar as used in this paper refers to "submicroscopic" perthite as well as ordinary microperthite.

color in the schillerized feldspars, fragments one centimeter in length were treated. The samples were heated in a platinum furnace and temperatures were measured with platinum-platinum 10% rhodium thermocouples. The temperatures were maintained at $\pm 5^\circ$ C. After each run the specimens were chilled in air and examined microscopically.

DESCRIPTION OF SPECIMENS

The composition and optical data for the specimens studied are given in Table 1.

TABLE 1. POTASH-SODA FELDSPARS

Specimen	K ₂ O	Na ₂ O	CaO	Or	Ab+An	α	β	γ	2V
				weight, per cent					
1. Orthoclase-microperthite, ¹ New Hampshire	12.61	2.38	0.16	78	22	1.5206	1.5246	1.5267	70°
2. Orthoclase-microperthite, ² California (El 38-265)†	12.10	2.47	n.d.	77	23	1.5205	1.5245	—	65°
3. Orthoclase-microperthite, ² California (El 38-167)	10.62	3.48	n.d.	68	32	1.5210	1.5250	—	65°
4. White moonstone, Ceylon	—	—	—	65	35*	1.5220	1.5260	—	70°
5. Blue moonstone, Ceylon	—	—	—	65	35*	1.5217	1.5257	—	70°
6. Blue moonstone, New Mexico	—	—	—	50	50*	1.5210	—	1.5270	37°
7. Blue moonstone, ³ New Mexico (JL7)	6.56	6.55	0.40	40	60	1.5220	—	1.5280	42°
8. Homogeneous sanidine, ³ Colorado (U3027)	10.35	4.26	n.d.	63	37	1.5200	—	1.5255	34°

* Composition estimated from optical properties.

† Numbers in parentheses refer to the numbers used in Larsen's collections

¹ F. A. Gonyer, analyst.

² Analyses from Larsen (personal communication).

Analyses from Larsen and others (6).

Specimen No. 1 is an orthoclase-microperthite which occurs as porphyroblasts in paragneisses of the Littleton formation in the Lovewell Mountain quadrangle, New Hampshire. The microperthitic structure is very uniform and the soda feldspar lamellae, as observed on (010), average about 0.04 millimeter in length.

Specimen No. 2 is an orthoclase-microperthite from the Rubideaux granite of the southern California batholith. The lengths of the soda feldspar lamellae as observed on (010) are variable, ranging from 0.05 millimeter to 0.25 millimeter.

Specimen No. 3 is an orthoclase-microperthite from the Rubideaux granite of the southern California batholith. The lamellae are irregular on (010) and range in length from 0.05 millimeter to 0.5 millimeter.

Specimen No. 4 is a white schillerized moonstone from Ceylon. It occurs in a sheared quartz-orthoclase rock which is "an acid phase of a pegmatite" according to Coates (4). The specimen shows conspicuous white schiller and a fine uniform microperthitic structure. The individual lamellae are fairly sharp and rarely exceed a few hundredths of a millimeter in length.

Specimen No. 5 is a blue schillerized moonstone from Ceylon and has the same mode of

occurrence as specimen No. 4. It exhibits an intense sky-blue schiller but appears homogeneous under the microscope.

Specimen No. 6 is a blue schillerized moonstone which occurs as phenocrysts in rhyolites of the Valles Grande Mountains, New Mexico. No definite microperthitic structure was seen under the microscope.

Specimen No. 7 is a blue schillerized moonstone which occurs as phenocrysts in rhyolites in the same area as specimen No. 6. The moonstone has a vague microperthitic structure.

Specimen No. 8 is a sanidine which occurs as phenocrysts in rhyolites in the Uncompahgre quadrangle, Colorado. The specimen exhibits no schiller or microperthitic structure.

RESULTS OF HEAT-TREATMENT

The more significant results of the thermal experiments on the orthoclase-microperthites are given in Table 2. In specimen No. 1 no change in the microperthitic structure was observed after prolonged heating at 700° C., but the refractive indices decreased about 0.001. A special set of experiments were run at 700° C. to determine the length of time necessary for the refractive indices to establish equilibrium. As shown in Fig. 1, the indices decreased appreciably in 13 hours at 700° C., but no further changes were noted after prolonged heating. At temperatures above

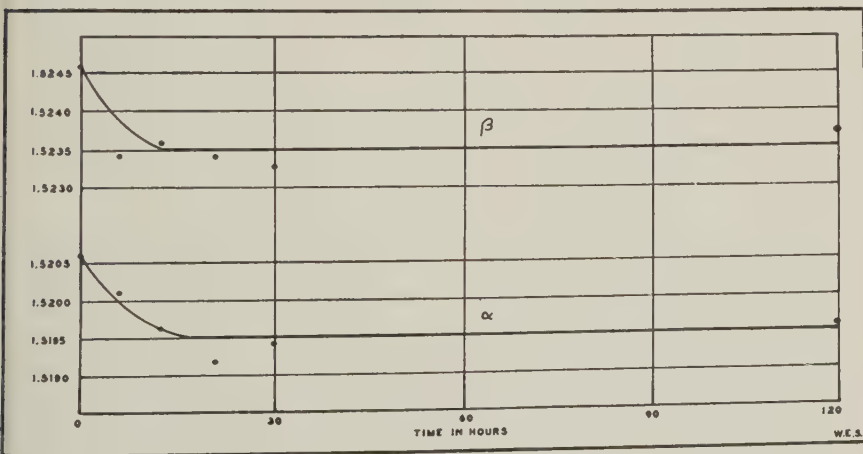


FIG. 1. Rate of change of the α and β indices of specimen No. 1 at 700° C.

850° C., numerous cracks developed in the specimen so that the refractive indices could not be accurately measured. After prolonged heating at 1000° C., the boundaries of the lamellae were found to be less sharp. On heating to 1075° C., the smaller lamellae disappeared and the larger lamellae became indistinct.

In specimens No. 2 and No. 3 of orthoclase-microperthite, no significant change in the microperthitic structure was observed on heating to

950° C. although the refractive indices decreased appreciably. These specimens were not studied at temperatures above 950° C.

TABLE 2. RESULTS OF HEAT-TREATMENT

Specimen No. 1, orthoclase-micropertthite (22% Ab+An), New Hampshire						
Specimen No. 2, orthoclase-micropertthite (23% Ab), California						
Specimen, No. 3, orthoclase-micropertthite (32% Ab), California						
Specimen	Temp. ° C.	Time in hrs.	α	β	2V	Micropertthitic structure
No. 1	22	—	1.5206	1.5246	70°	Lamellae 0.04×0.005 mm.
	700	192	1.5195	1.5237	70°	No apparent change
	900	100	*	*	70°	Boundaries slightly less distinct
	1000	300	*	*	40°	Slightly less distinct
	1040	370	*	*	35°	Lamellae smaller
	1075	330	*	*	20°	Only larger lamellae visible.
						Trace of glass
No. 2	22	—	1.5205	1.5245	65°	Fine and coarse lamellae
	950	19	1.5185	1.5223	65°	No significant change
No. 3	22	—	1.5210	1.5250	65°	Fine and coarse lamellae
	950	19	1.5184	1.5228	65°	No significant change

* Accurate index determinations not possible because of cracks in the heated material.

The results of the thermal studies of the moonstones which had large axial angles (specimens No. 4 and No. 5) are given in Table 3. In specimen No. 4, the micropertthitic structure was found to be less pronounced after heating to 800° C. Under ordinary light, the boundaries of the lamellae were indistinct and differences in relief were slight. Under crossed nicols, however, the lamellae were readily seen on favorably oriented grains. The fact that the white schiller was plainly visible after 145 hours, although somewhat fainter, indicated that the soda feldspar was only partially dissolved at 800° C.

The refractive indices decreased about 0.003 between 500° C. and 800° C. and showed little further decrease at higher temperatures. The decrease in index was as great after one hour at 600° C. as after 100 hours at the same temperature; therefore equilibrium was probably attained. As shown in Fig. 2, the total drop in indices does not occur at one definite temperature, but over a range of temperatures. On heating above 800° C., no further decrease in indices was observed although the micropertthitic structure and intensity of the schiller were appreciably reduced.

The study of blue Ceylon moonstone (specimen No. 5) also indicates

TABLE 3. RESULTS OF HEAT-TREATMENT
Specimen No. 4, white moonstone (35% Ab), Ceylon

Temp. ° C.	Time in hrs.	α	β	2V	Microperthitic structure	Schiller
22	—	1.5220	1.5260	70°	Fine lamellae	White schiller
400	42	1.5220	1.5260	70°	No apparent change	n.d.
500	90	1.5220	1.5260	70°	No apparent change	n.d.
600	116	1.5202	1.5247	70°	Lamellae slightly reduced	n.d.
800	6	1.5190	1.5232	70°	Lamellae indistinct under ordinary light. Fairly conspicuous under crossed nicols	n.d.
800	145	1.5190	1.5232	70°	No further change apparent	Less intense
900	138	1.5190	1.5230	70°	Faint	n.d.
990	18	1.5190	1.5230	70°	Faint	Very faint

Specimen No. 5, blue moonstone (35% Ab), Ceylon
(no microperthitic structure visible)

Temp. ° C.	Time in hrs.	α	β	2V	Schiller
22	—	1.5217	1.5257	70°	Intense blue
800	1	1.5190	1.5227	70°	Less intense
800	90	1.5190	1.5227	70°	Same as after one hour
1000	22	1.5190	1.5232	n.d.	Very faint
1000	700	1.5190	1.5232	40°	Same as after 22 hours

that the drop in refractive indices is completed before the final solution of the soda feldspar lamellae. Although the specimen appeared to be homogeneous under the microscope, the intense sky-blue schiller showed that a submicroscopic lamellar structure existed. After heating to 800° C., the refractive indices were found to be about 0.003 lower, and although the intensity of the schiller was reduced, a violet-blue color was still conspicuous. The schiller became very faint on heating to 1000° C. for 22 hours. No further change in the intensity of the schiller was observed after heating for 700 hours at 1000° C. and the mean refractive index was the same as after the run at 800° C.

The results of the heat-treatment of the moonstones which had small axial angles (specimens No. 6 and No. 7) are given in Table 4. In specimen No. 6, no definite microperthitic structure could be seen under the microscope. However, many of the lines in the x-ray powder photographs of the moonstone were diffuse and broad. These lines were doublets linked

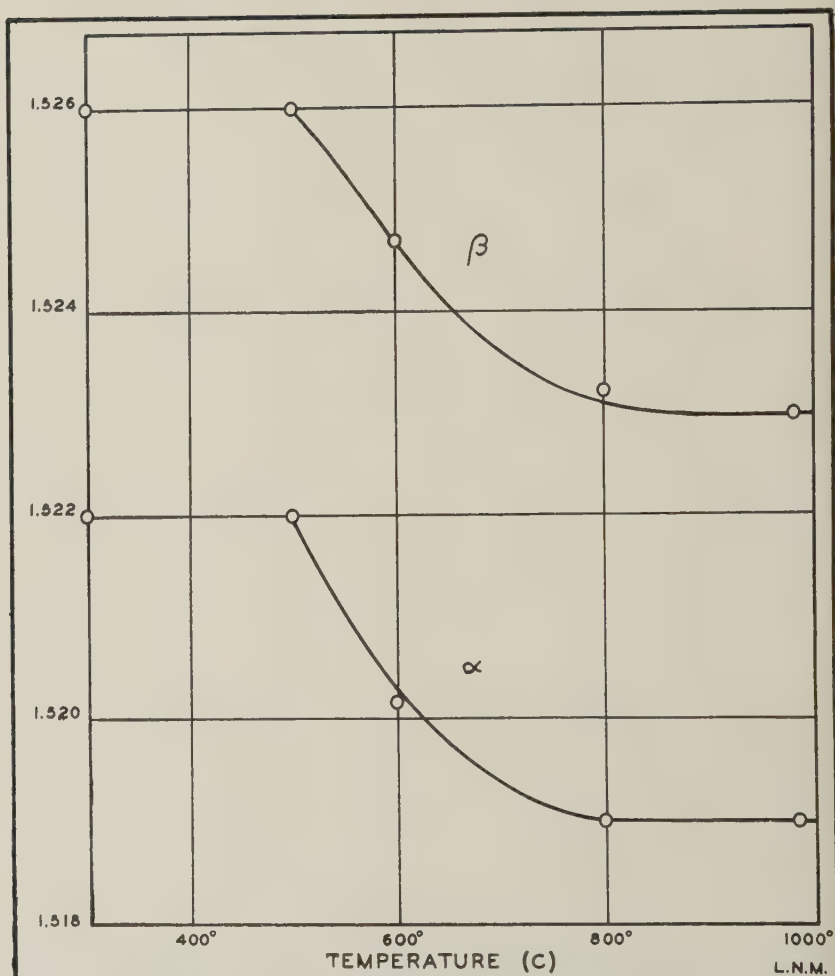


FIG. 2. Changes in the α and β indices of specimen No. 4 with changes in temperature.

by a band of blackening and were presumably due to the presence of two phases. On heating to 800° C. the blue schiller disappeared and the refractive indices decreased 0.0018. No further changes in indices were observed at temperatures up to 1000° C. In the x-ray powder photographs of the material that had been heated to 990° C., a single sharp line occurred in place of each doublet in the initial photographs. This indicated that homogenization had been completed.

In specimen No. 7 changes in the microperthitic structure were difficult to observe because the lamellae in the initial material were indistinct. On heating to 900° C., the lamellae apparently dissolved completely and

TABLE 4. RESULTS OF HEAT-TREATMENT

Specimen No. 6, blue moonstone (50% Ab), New Mexico

Specimen No. 7, blue moonstone (60% Ab-1-An), New Mexico

Specimen No. 8, homogeneous sanidine (37% Ab), Colorado

Specimen	Temp. ° C.	Time in hrs.	α	γ	2V	Remarks
No. 6	22	—	1.5210	1.5270	37°	Blue schiller. No visible microperthite
	700	48	1.5192	1.5252	37°	Very faint schiller
	800	96	1.5192	1.5252	37°	No schiller
	1000	80	1.5192	1.5252	—	No schiller
No. 7	22	—	1.5220	1.5280	42°	Vague microperthitic structure
	900	24	1.5200	1.5258	42°	No definite lamellae visible
No. 8	22	—	1.5200	1.5255	34°	Homogeneous
	800	24	1.5200	1.5255	34°	Homogeneous

the refractive indices decreased 0.0021. No attempt was made to determine the effects on the schiller in this specimen because the fragments available were so small that significant changes could not be observed.

Homogeneous sanidine (specimen No. 8) showed no decrease in refractive indices on heating to 800° C. for 24 hours (Table 4). Under similar treatment the microperthitic white moonstone (specimen No. 4) of approximately the same composition showed a 0.003 decrease in indices.

COMPARISON OF RESULTS WITH THOSE OF PREVIOUS WORKERS

Barth (1) found that orthoclase-microperthite of his "guttate" type, which contained 25% soda feldspar, became homogeneous by heating to 1000° C. for 700 hours. Kôzu and Endô (5) reported that Ceylon moonstone containing 26% soda feldspar became homogeneous between 1060° C. and 1115° C. Spencer (8), on the other hand, concluded that in orthoclase-microperthites containing less than 30% soda feldspar, homogenization was essentially completed at temperatures below 800° C.

The results of the present study indicating that temperatures of over 1000° C. are required to homogenize the low-soda perthites (specimens No. 1 and No. 2) are in better agreement with the earlier studies than with the more recent data of Spencer. Although the microperthitic structure in Spencer's specimens was finer than in the writer's, the duration of the runs in the present study appears to have been sufficient for the attainment of equilibrium. A possible explanation for the difference in be-

havior of Spencer's low-soda specimens is given in a later section.

The writer's determinations of the temperatures required to homogenize the high-soda lamellar feldspars ($>30\%$ Ab) are in good agreement with the results of Spencer and earlier workers.

FACTORS WHICH INFLUENCE THE TEMPERATURE AT WHICH LAMELLAR FELDSPARS BECOME HOMOGENEOUS

The results of the thermal studies of feldspars which have been conducted up to the present time indicate that the lamellar feldspars which have axial angles less than about 60° become homogeneous at temperatures below 850° C. In the lamellar feldspars that have axial angles greater than about 60° , temperatures above 1000° C. are required to effect homogenization.

The difference in behavior of these lamellar feldspars is probably due to differences in structure. Although the structural data on feldspars are meager, they do suggest that the lamellar feldspars which have large axial angles are structurally different from those with small axial angles. In Fig. 3, it is seen that in the lamellar feldspars studied by *x*-ray methods those with axial angles larger than about 60° correspond to the "M" structural type of Chao and Taylor (3), whereas those with smaller axial angles correspond to the "F" structural type.

This difference in structure may account for the apparent disagreement between the writer's results and those of Spencer in the specimens which contain less than 30% soda feldspar. Spencer's low-soda micropertthites had axial angles less than 60° and became homogeneous at temperatures below 800° C. In the writer's specimens the axial angles were greater than 60° and temperatures above 1000° C. were required to effect homogenization. Kôzu and Endô (5) also found that a temperature of over 1000° C. was necessary to homogenize their specimen of low-soda (Ceylon) moonstone which had an axial angle of 66° .

The change in axial angle which occurs before homogenization is completed is apparently unrelated to a change in structure which facilitates solution of the lamellae. Although the axial angle of specimen No. 5 was reduced from 70° to 40° at 1000° C., homogenization was not completed even after 700 hours at this temperature (Table 3).

RELATION BETWEEN AXIAL ANGLE AND DEGREE OF SOLUTION IN LAMELLAR FELDSPARS

It is generally accepted that in unheated lamellar feldspars which have relatively large axial angles no more than about 10% soda feldspar is held in solid solution. On the other hand, in the lamellar feldspars which have relatively small axial angles, Spencer (8) and Chao and Taylor (3) concluded that a large part of the soda feldspar was held in solid solution.

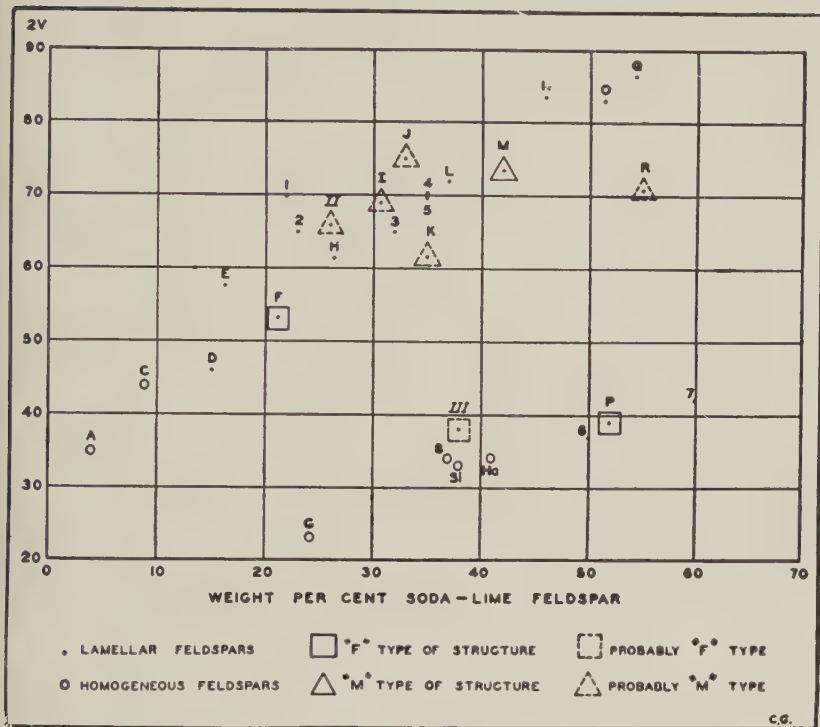


FIG. 3. Axial angles of potash-soda feldspars plotted against composition. Numbers correspond to specimens in Table 1. Letters refer to the specimens studied by Spencer (8). II and III refer to specimens studied by Kôzu and Endô (5). The structures of specimens F, M, and P were determined by Chao and Taylor (3). Structures of specimens I, J, K, and R based on preliminary *x*-ray studies by Chao and Taylor (3).

However, *x*-ray powder photographs of specimen No. 6, which has an axial angle of only 37°, did not indicate that an abnormal amount of soda feldspar was held in solid solution. The spacings of the potash feldspar lines in specimen No. 6 were found to be essentially the same as in the lamellar feldspars of corresponding composition which had large axial angles.

From the Laue photographs of the lamellar feldspars studied by Kôzu and Endô (5), there is no indication that the proportion of soda feldspar held in solid solution in lamellar feldspars is related to the size of the axial angles.

CAUSES OF THE DECREASE IN REFRACTIVE INDICES

The results of the present study do not support Spencer's contention that the decrease in refractive indices which occurs during the heating of

lamellar feldspars is related solely to solution of the soda feldspar lamellae. In the orthoclase-microperthites (specimens Nos. 1, 2, and 3), no significant decrease in the size of the lamellae was observed during the drop in indices. In the Ceylon moonstone (specimen No. 4), the microperthitic structure became less distinct in the same temperature interval during which the indices decreased, but the persistence of the schiller at still higher temperatures indicated that the solution was only partially completed. During the solution of the lamellae at higher temperatures, no further decrease in indices was observed. In Fig. 4, it is seen that

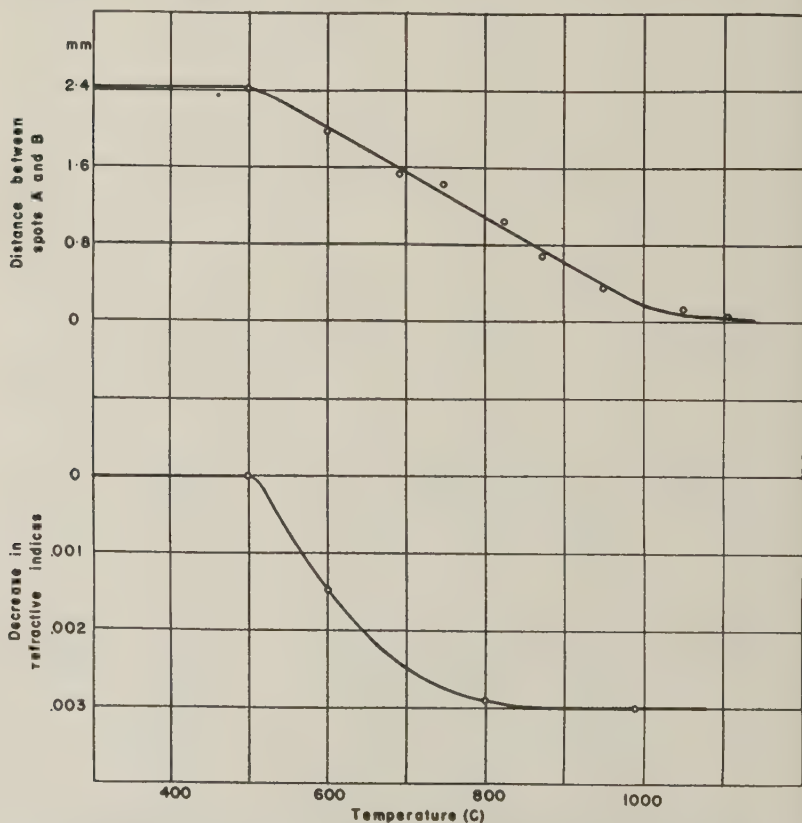


FIG. 4. Comparison of the rate of convergence of the two Laue spot systems in moonstone with the rate of decrease of the refractive indices on heating. The measurements were not made on the same sample of moonstone but the specimens compared were similar in the size of the axial angle, composition, and mode of occurrence.

Upper curve: Decrease in the distance between the two Laue spot systems of Ceylon moonstone as determined by Kôzu and Endô (5). Distance was measured between the two spots designated by Kôzu and Endô as A and B (p. 13).

Lower curve: Rate of decrease of the refractive indices of Ceylon moonstone, specimen No. 4.

the rate of solution of the soda feldspar lamellae in Ceylon moonstone as determined by the *x*-ray studies of Kôzu and Endô (5) is not the same as the rate of change of index as determined in the present study of Ceylon moonstone. The drop in refractive indices in the writer's specimen was completed well below the temperature at which the less sodic moonstone of Kôzu and Endô became homogeneous. For these reasons, it is doubtful whether the decrease in indices on heating is related only to solution of the lamellae.

An inversion of the soda feldspar component to a form with lower indices does not satisfactorily account for the observed facts. In the series of micropertthites which Spencer (8) studied, the drop in indices on heating to 850° C. was proportional to the soda content between 8 per cent and 30 per cent soda feldspar. With greater soda content the drop in indices was less. Therefore, an inversion of the soda feldspar component does not appear to be the sole cause of the decrease in indices.

As Buerger (2) has pointed out, the homogenization of perthite may be regarded as a transformation of two ordered crystals to one disordered crystal. He states that at the onset of disorder there is commonly a tendency for open spaces to develop. This increase in volume might account for part of the decrease in indices.

Until more is known about the structure of lamellar feldspars, it may not be possible to give a complete explanation of the changes in indices. Chao and Taylor (3) state that the structure of the soda feldspar component in lamellar feldspars may be different from that of ordinary albite. Furthermore, the structure of the soda feldspar component in the "F" type of lamellar feldspar may not be the same as that in the "M" type. With so many variables it is difficult to say what changes in volume take place on heating. The decrease in refractive indices may be due partly to solution of the lamellae and partly to inversions in one or both of the components of the lamellar feldspars. The amount of decrease may depend as much upon the structure of the feldspar as upon its bulk composition.

ORIGIN OF THE LAMELLAR FELDSPARS

The lamellar feldspars that have small axial angles become homogeneous at temperatures (below 850° C.) which are probably lower than the crystallization temperatures of many homogeneous feldspars. These lamellar feldspars, therefore, may be of exsolution origin.

The fact that high temperatures (above 1000° C.) are required to homogenize the lamellar feldspars which have large axial angles raises the question as to whether the soda feldspar lamellae in these specimens are entirely of exsolution origin. The writer has found that mixtures of quartz and sodic potash feldspar become largely liquid at 1000° C. under

atmospheric conditions. The lamellar feldspars which have large axial angles may be examples of anomalous mixed crystals (7). These intergrowths are formed by simultaneous intercrystallization of two phases, one being disseminated in oriented fashion in the host crystal as a sort of buried overgrowth.

SUMMARY

In many of the finely lamellar feldspars which have axial angles greater than 60° , the temperatures required to effect homogenization probably do not represent the minimum temperatures at which the feldspars could have crystallized. The lamellar feldspars with large axial angles may have formed largely by the simultaneous crystallization of potash feldspar and soda feldspar rather than by the exsolution of an initially homogeneous feldspar. The lamellar feldspars which have axial angles less than about 60° are probably of exsolution origin. The changes in refractive indices which accompany heating may be due partly to solution of the lamellae and partly to inversions of one or both of the components of the lamellar feldspar. The temperature at which the indices cease to decrease cannot be relied upon to give the temperature at which homogenization is completed. Some of the relatively fine lamellar feldspars in the low-soda group were found to be very resistant to heat-treatment compared to Spencer's specimens. The writer proposes that the specimens in the two cases may be structurally different and therefore do not respond similarly to thermal treatment. Data from x-ray investigations indicate that there is no significant difference between the degree of solution in the unheated lamellar feldspars which have small axial angles and those which have large axial angles.

ACKNOWLEDGMENTS

The writer is deeply indebted to Dr. George C. Kennedy for valued guidance in the experimental work and to Professor Esper S. Larsen for helpful suggestions during the progress of the investigation. Grateful appreciation is extended to Professors Marland P. Billings, Esper S. Larsen, and Clifford Frondel for critical reading of the manuscript. Specimens Nos. 2, 3, 6, 7, and 8 were kindly furnished by Professor Esper S. Larsen and specimens Nos. 4 and 5 were obtained from the mineralogical collections of the Department of Mineralogy and Petrography of Harvard University.

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COMPOSITION OF VEATCHITE¹

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AND

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ABSTRACT

Veatchite was described in 1938 as a hydrous calcium borate, $2\text{CaO} \cdot 3\text{B}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$, a lower hydrate of the colemanite-inoite series. Reinvestigation shows that it is a strontium borate with the probable formula $3\text{SrO} \cdot 8\text{B}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$.

INTRODUCTION

Veatchite was described by Switzer² as a new hydrous calcium borate, $2\text{CaO} \cdot 3\text{B}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$, from Lang, California. Shortly thereafter it was pointed out by Dr. W. T. Schaller³ of the U. S. Geological Survey that on the basis of his studies of the colemanite-meyerhofferite-inoite series⁴ the physical properties of veatchite were not consistent with its chemical composition, as given. It was at his suggestion, therefore, that veatchite was re-examined. A spectrographic examination of veatchite, kindly made by K. J. Murata of the U. S. Geological Survey, showed that the mineral is a strontium borate and not a calcium borate, as first reported. Veatchite is the only known naturally occurring strontium borate.

In a second find of veatchite, recently made at Lang, the mineral has a platy rather than a fibrous habit. It occurs in thin seams associated with colemanite in gray shale. Surfaces parallel to these seams are coated by a very thin layer of veatchite having a pronounced pearly luster.

Although veatchite is the first reported strontium borate, other strontium minerals are found in or near many of the Mohave Desert region borate deposits. Strontianite is found in deposits of commercial importance 10 miles north of Barstow, San Bernardino County, California.⁵ Celestite has been noted in the borate deposits at Borate, San Bernardino County, California; at Mt. Blanco, Death Valley, California, and at White Basin, Clark County, Nevada.

¹ Published by permission of the Secretary, Smithsonian Institution, and of the Director, U. S. Geological Survey.

² Switzer, George, Veatchite, a new calcium borate from Lang, California: *Am. Mineral.* **23**, 409-411 (1938).

³ Personal communication.

⁴ Schaller, W. T., Borate minerals from the Kramer district, Mohave Desert, California: *U. S. Geol. Survey, Prof. Paper* **158**, 169 (1930).

⁵ Knopf, Adolph, Strontianite deposits near Barstow, California: *U. S. Geol. Survey, Bull.* **660**, 257-270 (1917).

CHEMICAL ANALYSES

The original analysis of veatchite reported $\text{SrO} + \text{CaO}$ all as CaO , resulting in an incorrect formula being deduced. The spectrographic analysis, showing dominant strontium, also showed that a little calcium was present so that a new chemical analysis was required.

A new partial analysis of the type material (Table 1, column 1) was made on a sample very kindly furnished by Dr. Clifford Frondel of Harvard University. The analysis given in Table 1, column 3 was made

TABLE 1. CHEMICAL ANALYSES OF VEATCHITE

	1	2	3	4	5	6	7	8	9
SrO	29.5	29.7	29.8	30.3	31.08	31.20	30.0	$.317 = 3 \times .106$	32.45
CaO	1.7	1.7	1.3	1.4			1.6		
B_2O_3	57.3	57.8	57.7	58.6	59.03	59.26	58.5	$.840 = 8 \times .105$	58.16
$\text{H}_2\text{O}(+)$			9.6	9.7	9.50	9.54	9.6	$.533 = 5 \times .107$	9.39
Insol.	1.0				0.62				
			98.4	100.0	100.23	100.00	100.00		100.00

1. Veatchite, Lang, California. Fibrous habit. New partial analysis of type material. (Harvard Museum No. 92944 and U. S. National Museum No. 105697) W. W. Brannock, analyst.
2. Same as 1, recalculated to correct for insoluble.
3. Veatchite, Lang, California. Platy habit (U.S.N.M. R-8327). W. W. Brannock, analyst.
4. Same as 3, corrected for insoluble and calculated to 100%.
5. Veatchite, Lang, California. Original analysis. F. A. Gonyer, analyst.
6. Same as 5, corrected for insoluble and calculated to 100%.
7. Average of 2, 4 and 6.
8. Molecular ratios. Formula becomes $3(\text{Sr}, \text{Ca})\text{O} \cdot 8\text{B}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$.
9. Calculated composition for $3\text{SrO} \cdot 8\text{B}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$.

on the material of platy habit described above. Both new analyses were of necessity made upon 0.1 to 0.2 gram samples.

The chemical data are summarized in Table 1. The molecular ratios in best agreement indicate the probable formula to be $3(\text{Sr}, \text{Ca})\text{O} \cdot 8\text{B}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$. A possible alternate formula $\text{SrO} \cdot 3\text{B}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$ is obtained if the ratios $\text{SrO} = 1 \times .317$, $\text{B}_2\text{O}_3 = 3 \times .280$ and $\text{H}_2\text{O} = 2 \times .275$ are chosen. Additional analyses on larger amounts of material will be needed to demonstrate positively which formula is correct.

UNIT CELL DIMENSIONS

The unit cell dimensions were given by Switzer⁶ as $a_0 = 6.72 \text{ \AA}$, $b_0 = 41.26 \text{ \AA}$, $c_0 = 41.20 \text{ \AA}$. These values, though given as Angstrom units

were in reality in kX units. However, precision of the measurements is such that the difference is not significant. The best value of β is probably that of Murdoch⁷ who gives $\beta = 121^\circ 02'$.

Using the values given above and a unit cell content of $16[3\text{SrO} \cdot 8\text{B}_2\text{O}_3 \cdot 5\text{H}_2\text{O}]$, the calculated density becomes 2.586. This value compares favorably with the measured value 2.58.⁸

If the alternate formula $\text{SrO} \cdot 3\text{B}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$ is used and the unit cell content taken as $44[\text{SrO} \cdot 3\text{B}_2\text{O}_3 \cdot 2\text{H}_2\text{O}]$, the calculated density becomes 2.590.

⁶ *Op. cit.*

⁷ Murdoch, Joseph, Crystallography of veatchite: *Am. Mineral.*, **24**, 130–135 (1939).

⁸ Murdoch, Joseph, *op. cit.*

WHERRYITE, A NEW MINERAL FROM THE MAMMOTH MINE, ARIZONA*

JOSEPH J. FAHEY, *U. S. Geological Survey*; E. B. DAGGETT, *Richfield, Utah*; AND SAMUEL G. GORDON, *The Academy of Natural Sciences of Philadelphia*.

ABSTRACT

A new mineral from the Mammoth Mine, Arizona, having the formula $\text{PbCO}_3 \cdot 2\text{PbSO}_4 \cdot \text{Pb}(\text{Cl}, \text{OH})_2 \cdot \text{CuO}$ is named in honor of Dr. Edgar Theodore Wherry. The indices of refraction are $\alpha=1.942$, $\beta=2.010$ and $\gamma=2.024$, and $2V=50^\circ$ (calculated). The specific gravity is 6.45. Wherryite was found in a vug just above the 760-foot level associated with chrysocolla, diaboléite, and paralaurionite.

INTRODUCTION

In May of 1943 one of the authors, E. B. Daggett, then mining engineer at the Mammoth Mine, discovered a small vug of leadhillite crystals associated with cerussite, anglesite, phosgenite, paralaurionite, hydrocerussite, diaboléite, boléite, matlockite, and quartz. Within the cavity was some friable chalcocite with a relict structure of the galena which it has replaced.

The massive wall of the vug consisted of a light-green fine granular mineral enclosing some bluish chrysocolla, and at the cavity some blue diaboléite and greenish paralaurionite. This green matrix was up to five cm. in thickness and extended to the silicified wall of the vein—an altered quartz monzonite.

The minerals of this remarkable vug, and the crystallography of the leadhillite will be described in a later paper; some observations on the paragenesis will be given here in order to describe the occurrence of the green matrix which has proven to be a new mineral.

The name wherryite is given to this new mineral in honor of Dr. Edgar Theodore Wherry, first editor of the *American Mineralogist*, formerly curator of mineralogy at the U. S. National Museum, and now professor of plant ecology at the University of Pennsylvania.

LOCALITY

The Mammoth mine was named for the town of Mammoth, on the San Pedro River, which is about 3 miles to the northeast. It is in Pinal County, about 46 miles northeast of Tucson. An earlier post-office at the mine was called Schultz after the discoverer of the deposit. The present designation of the post-office is Tiger, so named by one of the owners

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of the mine in allusion to his alma mater. The locality should not be confused with the famous Mammoth Mine in the Tintic District of Utah. Discovered between 1879 and 1882, it has been worked at various times for gold, in World War I for the molybdenum content of the wulfenite, in 1933 for the vanadium content of the vanadinite and descloizite, and more recently for the lead, zinc, and silver values of the primary galena and sphalerite.

GEOLOGY AND ORE VEINS

The basement rock is a quartz monzonite, intruded by dikes of aplite and by bodies of andesite porphyry, now altered, as well as by later dikes and sills of rhyolite, and intrusive breccias of the earlier rocks.

The two important veins are the Mammoth vein and the Collins vein, which occupy premineral fissures with a general northwestward strike and a steep dip. Between them is the Mammoth fault, a brecciated zone about 20 feet in width. There can be little doubt that the Mammoth vein is the faulted apex of the Collins vein. The veins are thoroughly oxidized, with but few remnants of primary sulfides except in the lower levels. The approximate, but varying boundary between the oxides and the sulfides in the Collins vein lies between the 600-foot and 700-foot levels.

The leadhillite vug was found in the "hanging wall vein," a small block of ore which had been broken by the hanging wall fault from the Mammoth vein, and occurs between the Mammoth vein and the Mammoth fault, which in this part of the mine was more than 400 feet from the Collins vein. The vug was just above the 760-foot level, and close to the water table (Fig. 1).

Part of the paragenesis of the vug may be interpreted as follows: A mass of primary galena, close to the water table, was partly replaced by chalcocite in the course of secondary enrichment of the copper. Advancing oxidation altered some of the galena to anglesite, which was gradually replaced by cerussite, while some of the chalcocite was altering to chrysocolla. The sequence, galena to anglesite to cerussite, is the normal course of the weathering process.

At some stage of this normal sequence, lead sulfide to lead sulfate to lead carbonate, a remarkable reversal took place with the formation of complex sulfates with CO_2 and Cl_2 (leadhillite and wherryite), as well as the formation of the rare basic carbonate hydrocerussite, and the rare oxychlorides diaboléite, paralaurionite, and the chlorofluoride matlockite. It may be that this strange paragenesis was caused by the influx of solutions containing Cl ions in channels opened up by fault movements (perhaps formation of the Mammoth fault system).

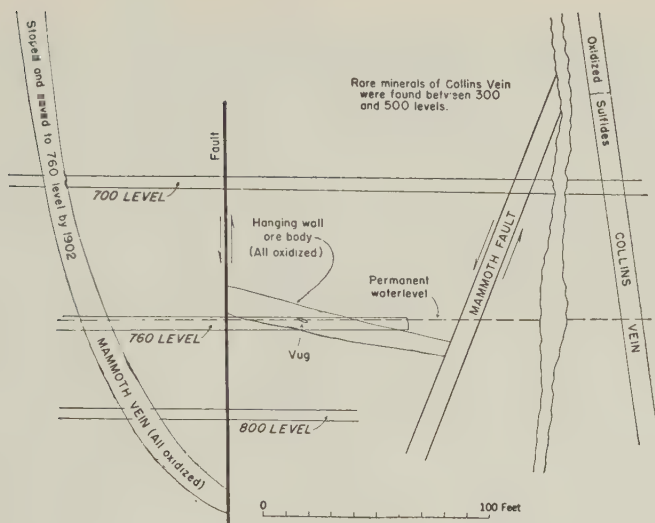


FIG. 1. Site of leadhillite vug in the hanging wall (faulted block) ore body in which wherryite was found.

OPTICAL PROPERTIES

Under crossed nicols most grains of wherryite showed no extinction, being made up of tiny particles, each with its own orientation. A few grains showed wavy extinction. A few more gave sharp extinctions and with these the indices of refraction were measured using sodium light and piperine iodide melts:

$$\left. \begin{array}{l} \alpha = 1.942 \\ \beta = 2.010 \\ \gamma = 2.024 \end{array} \right\} \text{all } \pm 0.005$$

$$2V = 50^\circ \text{ (Calculated).}$$

X-RAY ANALYSIS

X-ray powder diffraction photographs (Fig. 2) were made, using copper radiation and a nickel filter, of wherryite, caledonite, leadhillite, hydrocerussite, and cerussite. The dissimilarities are evident. The lines of the powder diffraction pattern of wherryite are listed in Table 1. Visual estimates of the intensities of the lines were made on a scale of 10, where 10 represents the intensity of the strongest line.

PHYSICAL AND CHEMICAL PROPERTIES

The color of wherryite matches Plate XVIII, Light Oriental Green in Color Standards and Nomenclature by Robert Ridgway. No variation

TABLE 1. PLANAR SPACINGS AND RELATIVE INTENSITIES OF THE DIFFRACTION LINES IN THE X-RAY POWDER SPECTRUM OF WHERRYITE

Spacing, Å.	Intensity	Remarks
4.77	3	
4.57	8	
4.19	1	
4.02	1	
3.52	2	broad
3.39	1	
3.28	1	
3.14	8	
3.05	10	
2.88	4	
2.79	1	
2.74	6	
2.67	1	
2.51	2	
2.45	2	
2.29	3	
2.22	2	
2.11	2	broad
2.05	2	
1.877	7	
1.800	1	
1.740	2	
1.719	2	
1.664	1	
1.642	1	
1.600	1	
1.575	1	
1.521	1	
1.441	2	
1.399	1	
1.382	1	
1.347	1	
1.334	1	
1.302	2	very broad
1.180	2	broad
1.133	1	
1.124	1	

in the color was noticed among the individual grains of the sample analyzed. The mineral has a fine granular texture and is usually intimately associated with leadhillite, paralaurionite, and chrysocolla. Paralaurionite is present as radial aggregates of acicular crystals with a slightly pearly-lustered cleavage; the leadhillite is found as tabular grains showing a basal cleavage.

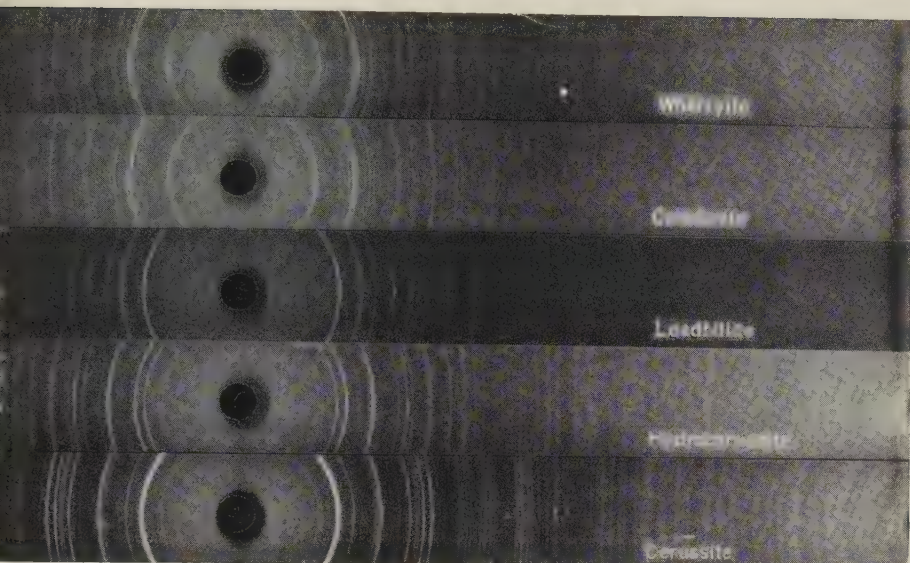


FIG. 2. X-ray powder diffraction photographs taken with copper radiation, nickel filter.

The specific gravity of the analyzed sample, determined with an Adams-Johnston pycnometer of fused silica, is 6.25. A correction for the 2.2 per cent quartz present raises the figure to 6.45, the specific gravity of wherryite.

Heated in a closed tube the mineral turns black without decrepitation and then melts.

It is slowly soluble in cold HCl (1:1) and in cold HNO_3 (1:1). Boiling H_2SO_4 (1:1) very slowly decomposes wherryite, leaving a white residue of PbSO_4 .

CHEMICAL ANALYSIS

The chemical analysis was made on 280 mg. of the 400 mg. sample. The 2.2 per cent that was insoluble in HCl was identified under the petrographic microscope as quartz. Carbon dioxide¹ was determined by measuring the volume of the gas. All other constituents were determined by the well-known methods outlined in Applied Inorganic Analysis by Hillebrand and Lundell. The analysis and computation of formulas are given in Table 2.

A spectrographic examination by K. J. Murata of the U. S. Geological

¹ Fahey, Joseph J., A volumetric method for the determination of carbon dioxide: *U.S. Geol. Survey, Bull.* **950**, 139-141 (1946).

TABLE 2. ANALYSIS AND FORMULA OF WHERRYITE

	Per cent		Ratios	
PbO	72.9	0.327	4.0	$\left\{ \begin{array}{l} 3.0 \text{ PbO} \\ 1.0 \text{ Pb} \end{array} \right.$
CuO	7.3	0.092	1.1	
SO ₃	13.0	0.162	2.0	
CO ₂	3.1	0.070	0.9	
Cl	0.9	0.025	0.160	2.0
H ₂ O-110° C.	none			
H ₂ O+110° C.	1.2 (OH)	0.135		
Insol.	2.2			
	100.6			
	-0.2 oxygen correction			
	100.4			

Formula: $\text{PbCO}_3 \cdot 2\text{PbSO}_4 \cdot \text{Pb}(\text{Cl}, \text{OH})_2 \cdot \text{CuO}$.

Survey showed Cu, Pb, and Si to be present in quantities greater than 1 per cent, Al in tenths of 1 per cent, Ca, Zn, and Mg in hundredths of 1 per cent and Fe and Na in thousandths of 1 per cent.

ACKNOWLEDGMENTS

The authors are grateful to many of their colleagues for help in preparing this paper. K. J. Murata of the U. S. Geological Survey made a spectrographic analysis, thereby establishing the order of magnitude of the minor constituents. Joseph M. Axelrod and Judith V. Weiss, also of the Survey Staff, made the x-ray powder photographs and measured the spacings. William Parrish of the North American Philips Co. made an x-ray spectrogram. The manuscript was greatly improved by the critical reading of Earl Ingerson, Michael Fleischer, and George T. Faust.

THE CRYSTAL STRUCTURE OF CRYPTOMELANE

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ABSTRACT

A crystalline specimen of cryptomelane, examined on a Weissenberg goniometer, is shown to be monoclinic with cell dimensions, $a=9.79 \text{ \AA}$, $b=2.88_6 \text{ \AA}$, $c=9.94 \text{ \AA}$, and $\beta=90^\circ 37'$, the space group being $Im(C_2^3)$ or $I2/m(C_{2h}^3)$. The formula KR_8O_{16} , R being mainly Mn^{4+} , corresponds well with chemical analysis.

Cryptomelane, a commonly occurring potassium manganese dioxide, was first identified as a distinct species by Ramsdell in his study of the manganese minerals (1). Although it may vary somewhat in composition, a formula KR_8O_{16} has been suggested (2), where R is mainly Mn^{4+} which is replaced to some extent by other metals, notably copper, zinc, cobalt, and Mn^{2+} . Crystalline occurrences are rare, but Weissenberg photographs taken on a cleavage specimen (3) and on fibrous needles (4) have given evidence of a body-centred tetragonal unit cell of dimensions: $a=9.82 \text{ \AA}$, $c=2.86 \text{ \AA}$. This conformed with both powder and fibre photographs (4).

We have recently examined a crystalline specimen (No. 4199) which was received from the Geological Survey of India by the courtesy of the Director, Dr. W. D. West. This was obtained from the Manganese Mine, Sitapar, Chhindwara District, Central Provinces and labelled hollandite. As the specimen contains 2.5% barium and 5% potassium and its powder diffraction pattern differs slightly from that of hollandite, it is considered to be cryptomelane. Fleischer and Richmond report the presence of cryptomelane in this area (2). It is now thought that cryptomelane and hollandite are isostructural (3, 5) and the barium is most probably present as an isomorphous replacement of the potassium.

The cryptomelane was composed of a matrix of small crystals up to 2 mm. in length, showing at least one good cleavage face, interspersed with parallel groups of crystals which had a tabular appearance, approximately 1 cm. square. Some haematite could be noted in a polished section. Specimens of the two types of crystals gave identical powder patterns which differed in detail from that of previous samples of cryptomelane (2). Many reflections, reported by Fleischer and Richmond as single, are resolved into doublets and triplets. The spacings derived from the proposed unit cell do not account for these and it would appear that the symmetry is lower than tetragonal. Comparison of the powder patterns for the Sitapar specimen with local massive cryptomelane samples

suggests that the small crystallite size of the massive mineral causes sufficient line broadening to obscure its lower symmetry. Difficulty was experienced in obtaining suitable crystals and even those which were selected for study were twinned. One crystal is related to its twin by a rotation of 90° and it is probable that the twinning occurs on the (100) and (001) faces, respectively. The twinning and close approximation to tetragonal symmetry indicate a similarity in the atomic distribution parallel to these faces. On zero-layer moving films taken on a Weissenberg goniometer (6), accurate spacings were measured and the symmetry established as monoclinic. The dimensions are $a=9.79 \text{ \AA}$, $b=2.88_6 \text{ \AA}$, $c=9.94 \text{ \AA}$ and $\beta=90^\circ 37'$. Volume of the unit cell $=280.7 \text{ \AA}^3$. The angle β was derived by triangulation using the spacings d_{100} , d_{101} and d_{101} . The

TABLE 1. ANALYSIS AND CELL CONTENTS OF CRYPTOMELANE (Sitapar)

Unit cell contents (O=16)				
MnO ₂	79.94%	Mn ⁴⁺	6.99	6.99
MnO	6.09	Mn ²⁺	0.65	1.01
Al ₂ O ₃	0.98	Al	0.15	
MgO	0.16	Mg	0.03	
Na ₂ O	0.74	Na	0.18	
BaO	2.76	Ba	0.14	1.10
K ₂ O	5.95	K	0.96	
H ₂ O+120° C.	0.36			
H ₂ O-120° C.	0.00			
Fe ₂ O ₃	3.42			
Total	100.40			
Density (obs.)	4.35	Density (calc.)		4.33

Absent: SiO₂, CaO, ZnO, CoO, WO₃ and P₂O₅.

The alkali contents were determined by Mr. C. E. S. Davis using the flame photometer.

radiation used was Fe K α ($\lambda=1.937 \text{ \AA}$). Indexing of moving films of $\{hk0\}$, $\{0kl\}$, $\{h0l\}$ and $\{h1l\}$ zones shows that reflections appear only when $h+k+l=2n$; the cell is body-centered and the space group Im or $I2/m$.

The chemical composition and computations to determine the contents of the unit cell are given in Table 1. For this purpose it is assumed that manganese may assume the valencies 4 and 2 and that all the Fe₂O₃ is present as a separate constituent.

The best formula which can be given is KRMn₇O₁₆, in which barium replaces potassium in the ratio 1:6. Gruner has suggested that sodium

may replace potassium (5), but the disparity in the ionic sizes has led to inclusion in R where R is Mn^{2+} , Al, Na and Mg. Since the formula corresponds to the contents of a body-centred unit cell, the formula for the equivalent group is $\text{K}_\frac{1}{2}\text{R}_4\text{O}_8$ (R being mainly Mn^{4+}). It would appear that only half of the potassium sites are occupied and no simple formula, e.g., $\text{K}_2\text{Mn}_8\text{O}_{17}$, containing two K ions per unit cell (as the limiting case) is compatible with the space group data, unless a certain proportion of Mn^{4+} passes to a lower valency state in order to maintain ionic balance. The potassium ions must be distributed throughout the structure in a random manner. The amount of potassium is not fixed and should be variable over a wide range without modification of the structure. Powder diffraction data from a laboratory preparation which was potassium free agree closely with that for cryptomelane, supporting this conjecture. The recorded analyses of cryptomelane (3) indicate that the potassium content does vary over a wide range. It suggests that this mineral is formed as a manganese dioxide mineral with intrusion of potassium ions, a sufficient change from Mn^{4+} to Mn^{3+} or Mn^{2+} occurring to preserve cationic balance. Some substitution by other metals for these lower valence ions may then occur.

A more detailed structure analysis of the Sitapar specimen is being carried out.

The work described in this report was carried out as part of the programme of the Division of Industrial Chemistry, Council for Scientific and Industrial Research, Australia.

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PHOSPHATE-ALLOPHANE IN AN EPIDOSITE FROM NORTH CAROLINA¹

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ABSTRACT

Phosphate-allophane has been found replacing oligoclase in an epidosite from North Carolina. A chemical analysis of the allophane and a petrographic description of the rock are given.

INTRODUCTION

An epidosite containing phosphate-allophane replacing feldspar was recently submitted to the Eastern Experiment Station, Bureau of Mines, for identification. The sample was found by J. H. Parrish, on his own property about 2 miles south of Whittier, North Carolina. Whittier is located in Swain County, where the Great Smoky Mountains of the Blue Ridge form a natural boundary between North Carolina and Tennessee. Epidosites and epidote-rich rocks are abundant throughout much of the Blue Ridge of North Carolina and Virginia. Unakite, a rock composed essentially of epidote, pink orthoclase and quartz was named by Bradley⁴ from its occurrence in the Unaka (Unicoi) Range of the Great Smoky Mountains bordering North Carolina and Tennessee. The Unaka Range lies a little southwest of Whittier.

Another unakite from Millam's Gap in the Blue Ridge of Virginia was described by Phelan.⁵ He describes this rock as being composed essentially of "old-rose feldspar (orthoclase) and green epidote." In both the North Carolina and Virginia occurrences of the unakites, the epidote is considered to be a secondary mineral. Johannsen,⁶ discussing the Virginia unakite, concurs, and suggests that the rock was derived from a hypersthene akerite by dynamic metamorphism and the percolation of meteoric waters.

CHEMISTRY

Phosphate-allophanes have been described by Ross and Kerr⁷ and more recently by Gordon.⁸ It has long been recognized that allophane is an

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⁴ Bradley, F. H., On unakite, an epidotic rock from the Unaka Range, on the borders of Tennessee and North Carolina: *Am. Jour. Sci.*, **VII**, 519-820 (1874).

⁵ Phelan, V. W., A new occurrence of unakite: *Smithsonian Miscell. Coll.* **XLV**, 306-316 (1904).

amorphous clay mineral or mineraloid having no definite crystalline structure or chemical composition. Ross and Kerr have indicated that allophane and the other mineraloids, evansite (hydrated aluminum phosphate), and chrysocolla (hydrated copper silicate) may occur as mixtures. These investigators have shown that mixtures of allophane and evansite may occur in all proportions. The previously ill-defined

TABLE 1. ANALYSIS OF PHOSPHATE-ALLOPHANES

	1	2	3	4
SiO ₂	25.00	27.61	21.26	17.76
Al ₂ O ₃	34.20	32.29	34.95	32.77
Fe ₂ O ₃	1.02	.23	.10	3.02
P ₂ O ₅	6.76	1.31	7.15	7.97
H ₂ O—	11.40	19.36	14.85	19.24
H ₂ O+	19.30	18.05	21.38	17.72
CuO	none	—	—	—
ZnO	none	—	—	—
MgO	tr.	.10	.05	.16
CaO	1.20	.02	none	.62
CO ₂	tr.	.72	—	—
SO ₃	none	.12	—	—
TiO ₂	tr.	none	—	—
Org. matter	—	—	tr.	—
Na ₂ O	tr.	}.10	—	—
K ₂ O	—		—	—
	98.88	99.91	99.74	99.26

1. Whittier, North Carolina, A. M. Sherwood, analyst.

2. Morehead, Kentucky, J. J. Fahey, analyst (Ross and Kerr⁷).

3. Huron, Indiana, J. J. Fahey, analyst (Ross and Kerr⁷).

4. Cerro de Llallagua, Bolivia, E. V. Shannon, analyst (Gordon⁸).

schroetterite and viterbite are also to be grouped with the allophanes. In all probability, phosphatic allophanes are more common than has been recognized, as the phosphorus is not always readily detected. In Table 1, the analysis of the phosphate-allophane from North Carolina is compared with others of similar composition. The separation of the analytical sample of the allophane from the epidote and feldspar in the

⁶ Johannsen, Albert, *A Descriptive Petrography of the Igneous Rocks: Vol. II*, 59–60, *Univ. of Chicago Press* (1939).

⁷ Ross, C. S., and Kerr, P. F., Halloysite and allophane: *U.S.G.S., Prof. Paper 185-G*, 144–148, (1934).

⁸ Gordon, S. G., The mineralogy of the tin mines of Cerro de Llallagua, Bolivia: *Proc. Acad. Nat. Sci. Phila.*, **XCVI**, 355, (1944).

North Carolina occurrence was difficult. It was finally accomplished by a combination of stage-grinding and high-intensity magnetic separation. A preliminary rough concentration was made of allophane+feldspar by selecting particles of the whitest material available. It was then determined that, with only a slight amount of grinding, the allophane, the softest constituent present, concentrated in the very fine screen fractions. The $-200+400$ mesh screen separation was composed entirely of allophane and fine grained epidote with a little sphene. The feldspar, because of superior hardness and coarser grain size, remained in the coarser screen fractions. After several passes at 1.5 amperes on the Franz Isodynamic Magnetic Separator, a small but microscopically clean allophane product was separated from the epidote and sphene. The complete chemical analysis was made on a one-half-gram sample.

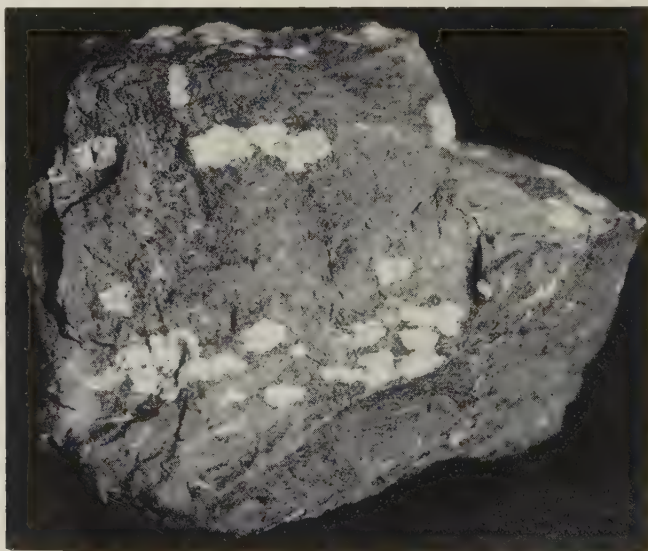


FIG. 1. Epidosite from North Carolina showing coarse-grained, oriented, altered oligoclase (white) in a matrix of fine-grained epidote. (Natural size.)

PETROGRAPHY

The North Carolina phosphate-allophane occurs as an alteration product of oligoclase in a weathered epidosite. The rock is not classified as a unakite because the feldspar in the type unakite is orthoclase rather than oligoclase. In a hand specimen, the rock is seen to consist of fine-grained, equigranular, green epidote sprinkled with medium- to coarse-grained, subhedral to euhedral, white insets of altered oligoclase (Fig. 1). The feldspar insets are oriented in the same manner as phenocrysts in a volcanic flow rock.

Thin-section studies show that the rock is composed of pale-green epidote (60–80%), oligoclase+allopahane+sericite (20–40%) and light brown sphene (1–5%). Calcite is sparingly present, associated with patches of sericite. A more detailed mode of the rock is unjustified, owing to the marked variation in different samples. Epidote, oligoclase, and sphene are subhedral to euhedral and all show a tendency toward a preferred orientation. Aggregates of sphene grains form narrow, parallel islands in the otherwise monomineralic epidote matrix. Several of the



FIG. 2. Phosphate-allophane (narrow strips) replacing oligoclase along twin lamellae to produce rectangular lattice dissection patterns. (Plane-polarized light, $\times 70$.)

large oligoclase insets show albite twinning; others show none. The feldspars commonly contain small euhedral inclusions of epidote and may be penetrated by larger peninsulas of epidote aggregates. Oligoclase is the only altered mineral and may be replaced by phosphate-allophane, sericite, or mixtures of sericite and a little calcite. The chief alteration product, however, is a colorless, glassy phosphate-allophane. It usually replaces the plagioclase along twin or cleavage planes forming rectangular lattices within the former (Fig. 2). In other instances the allopahane forms wormy streaks embaying the feldspar. All degrees of alteration may be observed, beginning with (a) peripheral replacement, passing through (b) rectangular lattice dissection patterns into (c) almost complete alteration, leaving only the original outlines of the plagioclase insets (Fig. 3). The average grain diameter of the epidote and sphene is 0.04 mm.; a maximum of 0.2 mm. was recorded for the former. The large insets of

plagioclase attain maximum diameters of three-eighths of an inch.

The absence of phosphate minerals other than allophane is unusual. Gordon⁸ found vivianite, wavellite, and vauxite associated with the phosphate-allophane from Cerro de Llallagua, Bolivia. He states that the allophane "was found interlaminated with marcasite, pyrite and sphalerite and probably derived from the weathering of pyrrhotite." Ross and Kerr⁷ do not state whether any phosphate minerals were associated with the phosphatic allophanes from Kentucky and Indiana.

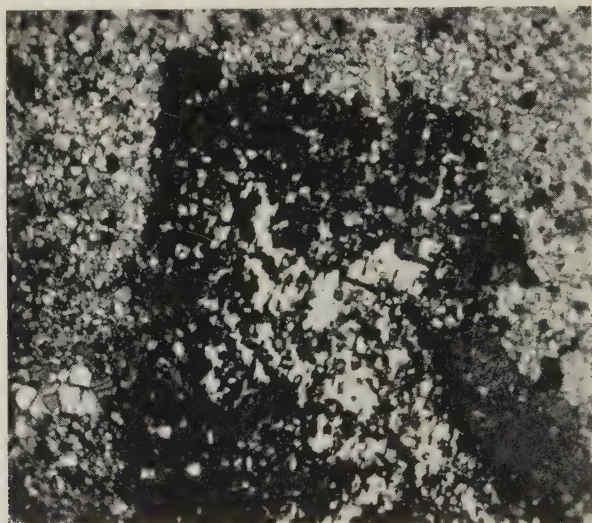


FIG. 3. A large grain of oligoclase (white) almost completely replaced by phosphate-allophane (black). The pseudomorph is bounded by fine-grained, granular epidote. (Crossed nicols, $\times 25$.)

The phosphate-allophane from Indiana, however, occurs in intimate association with halloysite ("indianite") and alunite and may have had an origin similar to the *Bolivian* material. Ries⁹ attributed the origin of the "indianite" to the decomposition of pyrite, the subsequent action of ground waters containing sulfuric acid on aluminous materials such as shale, and finally the replacement of quartz by halloysite, allophane, and alunite. The presence of lead, copper, iron, and zinc in allophanes associated with ore deposits is further testimony to the importance of the oxidation of sulfides in the formation of allophanes. Ross and Kerr⁷ make no mention of the origin of the phosphate found in several of the allo-

⁹ Ries, Heinrich, High-grade Clays of the Eastern United States: *U.S.G.S., Bull.* 708, 156 (1921).

phanes. Gordon⁸ does not discuss the matter either, but inasmuch as the Cerro de Llallagua tin deposits are very rich in phosphate minerals, the sources of phosphorus does not pose a problem. In the case of the North Carolina occurrence of phosphate-allophane, insufficient field data do not permit much theorizing on the origin of the material; however, the authors believe that the action of underground waters containing sulfuric and phosphoric acids was responsible for the alteration of the oligoclase to a phosphate-allophane. The sulfides, pyrrhotite, pyrite and chalcopyrite are abundant in Swain County, North Carolina. Their oxidation by meteoric waters may have been the source of circulating groundwaters carrying the sulfuric acid necessary for the alteration of feldspar to allophane.

ACKNOWLEDGMENT

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THE DIFFERENTIAL THERMAL ANALYSIS OF CERTAIN PHOSPHATES

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ABSTRACT

Fourteen samples of phosphates were studied by differential thermal analysis, x-ray, and petrographic methods. Minerals such as variscite, bolivarite, lazulite, wavellite, evansite, bobierite, vivianite, and zepharovichite (?) give endothermic reactions at low temperatures which indicate the loss of water of crystallization. Smaller endothermic reactions at higher temperatures are correlated with loss of hydroxyl, or possibly fluorine. Recrystallization on heating with the formation of aluminum phosphotridymite, aluminum phosphocristobalite, or berlinite (AlPO_4 with α -quartz structure) structures is common for the hydrous aluminum phosphates.

INTRODUCTION

The method of differential thermal analysis supplies data not easily obtained by ordinary dehydration studies. The most important data pertain to the dissociation and inversion phenomena that various phosphates undergo on being heated rapidly. The number of individual species investigated was limited by the material available which had been previously investigated by chemical or x-ray methods. Of the fourteen specimens here reported, the analyzed samples of only two were available. The identity of the remaining twelve samples was established by x-ray and petrographic examinations.

APPARATUS

The differential thermal analysis unit used in this investigation is similar to others described in the literature; the principal difference is in the removable sample block designed by J. W. Gruner (Fig. 1). The sample block is made of "inconel" metal rather than of the usual nickel.

DESCRIPTION AND BEHAVIOR OF SAMPLES

Variscite.— $\text{AlPO}_4 \cdot 2\text{H}_2\text{O}$, from cherty limestone, Fairfield, Utah. Light green prismatic grains which become lavender on heating slowly to 300°C . $G = 2.52$, $\alpha = 1.565$, $\gamma = 1.595$. A chemical analysis is not available, but the x-ray pattern is very similar to that for variscite from other localities. Petrographic examination shows the material to be homogeneous. Variscite (Fig. 2, no. 4) gives only one broad endothermic reaction with a peak at 260°C . The sample heated above 260°C . is still crystalline, but the structure developed has not been identified. A second structure, that of aluminum phosphotridymite (AlPO_4 with tridymite structure) is developed upon heating to 1000°C ., but there is no indication of this change in the differential thermal curve.

Bolivarite.— $\text{Al}_2\text{PO}_4(\text{OH})_2 \cdot n\text{H}_2\text{O}$, Ponte Verde, Spain, U. S. National Museum specimen R5594. Light gray-green powdery crust; isotropic, $N=1.493$. Amorphous to x -rays. No chemical analysis. A strong broad endothermic reaction with a peak at 220°C . (Fig. 2, No. 5) is the initial water loss. A sharp secondary endothermic reaction at 430°C . is inter-



FIG. 1. Detail of removable sample block.

preted as loss of the hydroxyl. Recrystallization is indicated by a weak exothermic reaction at 950°C ., and the final sample gave a faint x -ray pattern of aluminum phosphocristobalite.

Lazulite.— $\text{R}''\text{Al}_2(\text{OH})_2\text{P}_2\text{O}_8$, Graves Mountain, Georgia. Azure-blue monoclinic crystal. $\alpha=1.604$, $\beta=1.633$, $\gamma=1.639$, all $\pm .005$. Pleochroic; X colorless; Y and Z , azure-blue. X -ray pattern practically identical with lazulite from Matterhorn, Switzerland.

Lazulite (Fig. 2, no. 6) gives a moderate endothermic reaction with a peak at 220°C ., and a second strong endothermic reaction at 780°C . The final reaction is an exothermic peak at 925°C . The final heated product shows an aluminum phosphocristobalite structure with some earlier berlinite structure still showing.

Wavellite.— $\text{Al}_3(\text{OH})_3\text{P}_2\text{O}_8 \cdot 5\text{H}_2\text{O}$, Montgomery, Arkansas. Light green spherulites. Gives water in closed tube. $\alpha=1.535$, $\beta=1.540$, $\gamma=1.553$; all ± 0.003 . No chemical analysis; x -ray pattern is very similar to that reported for wavellite by McConnell (1942).

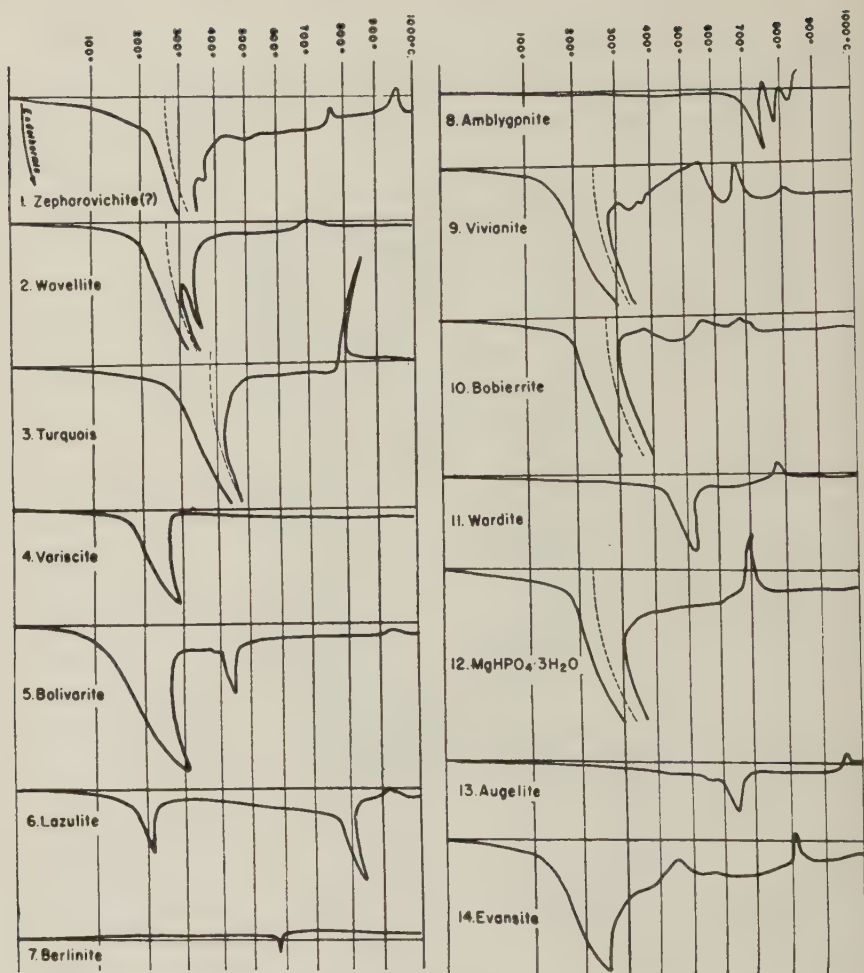


FIG. 2. Differential thermal curves. (Dashed arcs give temperature of broken peaks)

Wavellite (Fig. 2, No. 2) shows a strong endothermic reaction at 275°C . followed immediately by a second smaller endothermic peak at 315°C . An exothermic reaction is developed at 710°C . which may be related to recrystallization with the formation of aluminum phosphotridymite. A second sample of wavellite from an unknown locality shows the various reactions described above, but an additional exothermic peak is developed at 770°C . which is more pronounced than the exothermic reaction at 710°C . When cooled and x -rayed this second sample

also showed a phosphotridymite structure.* The optical properties of the second sample of wavellite did not differ appreciably from the first sample, but a closed tube test showed the presence of fluorine in the mineral, and possibly this element is responsible for the variation in the differential thermal curve.

Evansite.— $3\text{Al}_2\text{O}_3 \cdot \text{P}_2\text{O}_5 \cdot 18\text{H}_2\text{O}$, Goldberg, Idaho, U. S. National Museum specimen #86180. A brown resinous crust. Isotropic, $N=1.483$. Amorphous to x -rays, the sample was identified by optical means. Evansite (Fig. 2, No. 14) gives a large endothermic reaction with a peak at 190°C . A second endothermic reaction at approximately 350°C . and a third at approximately 525°C . are poorly defined. Recrystallization takes place at 840°C . as indicated by a well-developed sharp exothermic reaction. The structure at 1000°C . is phosphocristobalite.

Bobierrite.— $\text{Mg}_3(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$, from a fossil mammoth tusk from a gravel pit near Edgerton, Minnesota (Gruner 1943). White prismatic aggregate. $G=2.2$, $\alpha=1.51$, $\beta=1.52$, $\gamma=1.543$. The sample gives an x -ray pattern very similar to that of pure $\text{Mg}_3(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$ (Eimer and Amend Chemical Co.). A dehydration curve for bobierite is shown in Fig. 3. The sample was heated and weighed at intervals of 50°C . A part of the sample was removed at temperatures of appreciable water loss for x -ray study. Bobierite (Fig. 2, no. 10) gives a strong endothermic reaction with a peak at 280°C . at which temperature the mineral loses five molecules of water. A broad secondary endothermic reaction with a peak at 470°C . indicates the loss of the remaining three molecules of water. The x -ray pattern of the sample after heating to 300°C . shows little loss of structure although 60 per cent of the water has been removed; however, on heating to 470°C . with loss of three remaining molecules of water, the structure is destroyed. Recrystallization to $\text{Mg}_3(\text{PO}_4)_2$ takes place at 565°C . as is also indicated by a sharp exothermic reaction. Two lesser exothermic reactions are recorded at 675°C . and 710°C .

There are important differences between the static dehydration curve and the differential thermal curve. The static dehydration curve (Fig. 3) shows that five molecules of water are lost between 100 – 150°C .; two molecules of water are lost between 150 – 350°C .; and the final molecule of water is lost above 350°C . Similar results were obtained by earlier investigators. De Schulten (1903) states "das Hydrat $\text{Mg}_3(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$ gibt bei 120° fünf Moleküle Wasser ab." Klement (1936) found that two

* The distinction between tridymite and cristobalite structures is difficult in several of the films. The most intense lines of the two structures are almost identical. In many cases if heating is continued long enough at 1000°C . the earlier tridymite structure will be followed by a cristobalite one. If such a change is arrested too early both structures will show in the film and cause confusion especially since the lines are diffused.

molecules of water were lost at 300° C. and the last molecule at 400° C. The differential thermal curve shows an endothermic reaction with a peak at 280° C. corresponding to a loss of five molecules of water and a second peak at 450° C. for the remaining three molecules of water. The difference of behavior of the mineral is the result of the rate of heating; rapid heating in the differential thermal analysis apparatus gives two breaks; whereas only one distinct break is shown on slower heating in the static dehydration method.

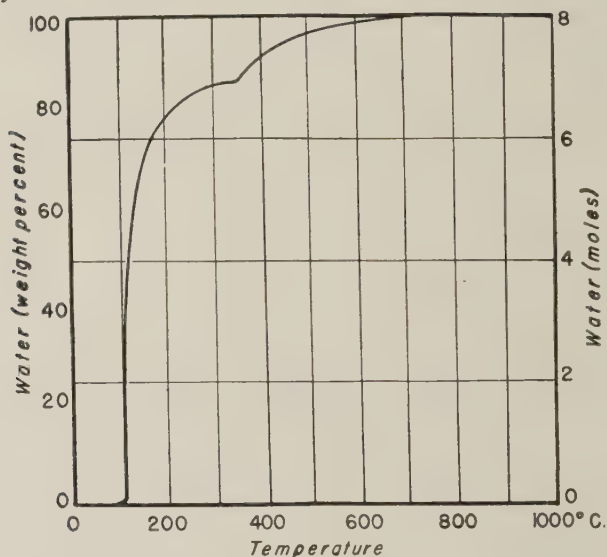


FIG. 3. Dehydration curve.

Vivianite.— $\text{Fe}_3(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$, Keystone, South Dakota. A medium blue, darkening after grinding to a powder. $G=2.56$. $\alpha=1.580$, $\beta=1.605$, $\gamma=1.635$. Strongly pleochroic: X = dark blue, Y =pale yellow, Z =olive green. The structures of bobierite and vivianite are closely related as shown by x -ray patterns.

Vivianite (Fig. 2, no. 9) shows a strong endothermic reaction with a peak at 260° C. corresponding to the loss of five molecules of water in bobierite. A second endothermic reaction is smaller with a peak at 330° C. during which two molecules of water are lost. The last molecule of water gives a small endothermic reaction with a peak at 380° C. The amplitudes of the endothermic peaks are roughly proportional to the water loss. A poorly defined exothermic reaction takes place at 550° C. This reaction is related to the oxidation of ferrous iron. Differential thermal curves for vivianite are hard to reproduce because of the ferrous

iron which is easily oxidized. Prolonged grinding may result in oxidation of sufficient ferrous iron to change the curve. Studies of minerals such as vivianite probably are best made in an atmosphere of an inert gas.

Augelite.— $\text{Al}_2\text{PO}_4(\text{OH})_3$ from Laws, California, U. S. National Museum, specimen #97576. Cream colored crystalline crust. $G=2.7$. Biaxial (+), $\alpha=1.575$, $\gamma=1.588$; both $\pm .003$. The x -ray pattern is similar to one published by Peacock (1941) for augelite from White Mountain, California, and the optical data are essentially the same.

Augelite (Fig. 2, no. 13) gives a strong endothermic reaction with a peak at 680°C . This supports the conclusion of Peacock that augelite contains hydroxyl. There is no endothermic reaction at a low temperature which might indicate the presence of water of crystallization. A sharp exothermic peak is recorded at 970°C . After heating the residue of augelite to 1000°C . a structure is developed as shown by x -ray patterns, which is unlike any of the aluminum phosphates noted previously.

Wardite.— $\text{CaNa}_4\text{Al}_{12}(\text{PO}_4)_8(\text{OH})_{18}\cdot 6\text{H}_2\text{O}$, Lewiston, Utah. A light gray-green dull mineral in nodules resembling turquoise in appearance. $G=2.78$. Tetragonal: $\epsilon=1.600$, $\omega=1.591$. The optical constants are essentially the same as those given by Larsen (1942) for wardite from Fairfield, Utah. Wardite (Fig. 2, no. 11) gives a strong endothermic reaction with a peak at 510°C ., and a moderate exothermic reaction with a peak at $780\text{--}800^\circ\text{C}$. X -rays show recrystallization to aluminum phosphocrystalite.

Turquoise.— $2\text{Al}_2\text{O}_3\cdot\text{P}_2\text{O}_5\cdot 5\text{H}_2\text{O}$, New Mexico. Dull-azure-blue, highly fractured. The x -ray pattern is comparable to that of other samples of turquoise. A very sharp endothermic reaction with peak at 380°C . and an extremely sharp exothermic reaction with peak at 840°C . are characteristic of the relatively simple differential thermal curve of turquoise (Fig. 2, no. 3). The residue shows an aluminum phosphocrystalite structure. Not all of an earlier berlinite structure has disappeared but probably would on longer heating.

Amblygonite.— $\text{LiAl}(\text{F}, \text{OH})\text{PO}_4$, Black Hills, South Dakota. A bluish-white cleavable mass. $G=3.0$. Biaxial positive. $\alpha=1.605$, $\gamma=1.627$. The optical data (Winchell, 1926) indicate a composition of amblygonite $\text{LiAl}(\text{F})\text{PO}_4$, 20 per cent and montebrasite $\text{LiAl}(\text{OH})\text{PO}_4$, 80 per cent. Amblygonite (Fig. 2, no. 8) gives an endothermic reaction with a peak at 740°C . followed by an exothermic reaction with a peak at 760°C . A second endothermic peak occurs at 780°C . which is followed by a second exothermic reaction at 800°C . The sample fused at 850°C .

Zepharovichite (?).— $\text{AlPO}_4\cdot 3\text{H}_2\text{O}$, Bohemia, U. S. National Museum specimen #84339. Grayish-white powder. $G=2.34$. $\alpha=1.525$, $\gamma=1.544$,

$\pm .003$. The sample gives a sharp endothermic reaction at 270° C. with a second endothermic reaction at 355° C. There are two exothermic (Fig. 2, no. 1) reactions; the first with a peak at 790° C., and a second at 970° C. The *x*-ray pattern of the heated residue shows aluminum phosphocristobalite with some phosphotridymite.

Zepharovichite is not a valid mineral species. Winchell (1927) noted the similarity of the optical data to that for wavellite, and McConnell (1942) pointed out that the *x*-ray pattern of zepharovichite is almost identical with that of wavellite. These findings are supported by the differential thermal curve; the temperatures of the endothermic peaks for

Name & Composition		Temperature									
		100	200	300	400	500	600	700	800	900	1000° C.
Wavellite	$\text{Al}_2(\text{OH})_2\text{P}_2\text{O}_5 \cdot 5\text{H}_2\text{O}$			V	V				Λ	Λ	
Zepharovichite	$\text{AlPO}_4 \cdot 3\text{H}_2\text{O}$			V	V				Λ		Λ
Bolvirite	$\text{Al}_2\text{PO}_4(\text{OH})_2 \cdot n\text{H}_2\text{O}$		V		V						Λ
Variscite	$\text{AlPO}_4 \cdot 2\text{H}_2\text{O}$			V							
Lazulite	$\text{R}^+\text{Al}_2(\text{OH})_2\text{P}_2\text{O}_5$		V						V		Λ
Turquoise	$2\text{Al}_2\text{O}_3 \cdot \text{P}_2\text{O}_5 \cdot 5\text{H}_2\text{O}$				V					Λ	
Evansite	$3\text{Al}_2\text{O}_3 \cdot \text{P}_2\text{O}_5 \cdot 18\text{H}_2\text{O}$		V							Λ	
Bobierite	$\text{Mg}_3(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$			V		V	Λ	Λ	Λ		
Vivianite	$\text{Fe}_3(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$			V	V	V	Λ	Λ		Λ	
$\text{MgHPO}_4 \cdot 3\text{H}_2\text{O}$			V					Λ			
Augelite	$\text{Al}_2\text{P}_2\text{O}_7(\text{OH})_2$							V			Λ
Wardite	$\text{CaNa}_2\text{Al}_2(\text{OH})_4(\text{PO}_4)_2 \cdot 6\text{H}_2\text{O}$					V			Λ		
Amblygonite	$\text{LiAl}(\text{F},\text{OH})\text{PO}_4$							V	Λ	V	
Berlinite	AlPO_4						V				

Symbols: (V) = Endothermic; (Λ) = Exothermic

FIG. 4. Summary of differential thermal data.

zepharovichite agree with those for wavellite, and the first exothermic reaction is found in wavellite. The second exothermic reaction at 970° C. was not recorded for wavellite.

Berlinite. — AlPO_4 , synthetic, milky-white crystals. $G = 2.56$. (Na) $\epsilon = 1.535$, $\omega = 1.525$, $\pm .002$. In the *x*-ray pattern all the lines are very similar to quartz, except d 1.380 and d 1.375 where the intensities of the berlinite lines are the reverse of the quartz lines.

Berlinite (Fig. 2, no. 7) shows a single sharp exothermic reaction at 585° C. which marks the inversion from the alpha to beta form. The inversion point is 10° C. higher than the inversion of alpha to beta form of quartz recorded on the same instrument.

$\text{MgHPO}_4 \cdot 3\text{H}_2\text{O}$. — (dibasic magnesium phosphate), Mallinkrodt Chemical Co. A fine-grained white powder. $G = 2.10$. $\alpha = 1.514$, $\gamma = 1.533$, $\pm .003$.

Dibasic magnesium phosphate (Fig. 2, no. 12) gives a strong endothermic reaction with a peak at 255° C. An exothermic reaction which begins at 620° C. is culminated in a peak at 680° C.

Certain generalizations are drawn from the present study, but as the available data are few, revisions may be necessary with the study of more samples. The differential thermal reactions of the fourteen samples are shown in Fig. 4. In this chart the temperatures of the reaction peaks have been plotted to scale. The relative magnitude of the reaction is approximately indicated by the size of the symbol plotted.

ACKNOWLEDGMENTS

This problem was suggested by Professor J. W. Gruner, and the laboratory studies were made under his direction. Valuable assistance was received from Dr. S. S. Goldich and Mr. Lynn Gardiner. Five samples were obtained through the kindness of Dr. William Foshag of the U. S. National Museum.

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NOTES AND NEWS

THERMAL DECOMPOSITION OF ZINC SULFIDE POLYMORPHS

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Crystals or small cleavage pieces of the five known polymorphs¹ of zinc sulfide when heated in air for 16 hours at 800° C. were found by x -ray study to be converted in every instance to the ordinary form of zinc oxide (zincite). ZnO has a wurtzite-type structure (2H) based on hexagonal closest-packing of the oxygen atoms. It was hoped that higher polymorphs of ZnO would be derived from the corresponding ZnS forms, but this did not prove to be the case. With wurtzite-4H, 6H and 15R, the ZnO produced by thermal decomposition in all instances formed single-crystal pseudomorphs oriented parallel to the original crystal, as determined by x -ray precession photographs taken about $[10\bar{1}0]$ and rotation photographs taken about $[0001]$. The perfection of the orientation varied somewhat among different crystals of these polymorphs. Ordinary wurtzite (2H) and particularly the isometric polymorph, sphalerite, showed a relatively large amount of disorientation. Rotation photographs of oxidized cleavage fragments of these substances showed well-marked powder lines, although with diffuse intensity maxima thereon that indicated a considerable degree of parallel orientation of the ZnO crystallites to the $[0001]$ or $[111]$ axes of the original ZnS crystal. In the case of sphalerite the disorientation effect is enhanced by the presence of the non-parallel but symmetrically equivalent tetrahedral planes of nucleation.

Crystals of wurtzite-4H and 6H when heated in an atmosphere of H_2S in sealed tubes at 700° were not found to have undergone an irreversible structural change. A crystal of wurtzite-6H heated in H_2S at 1000° was found to have sublimed. The oxidized crystals are pale yellow-brown in color, with a bright luster. Under the microscope all of the oxidized pseudomorphs were seen to consist of parallel aggregates of minute crystallites that behaved optically as a uniaxial individual.

¹ Frondel, C., and Palache, C.: *Am. Mineral.*, **35**, 29-42 (1950).

THE NATURE OF "ARIZONITE"

J. L. OVERHOLT, G. VAUX, AND J. L. RODDA*

"Arizonite" having the composition $\text{Fe}_2\text{O}_3 \cdot 3\text{TiO}_2$ was described by Palmer (4) and frequently has been reported as a constituent of ilmenite black sands (1). Attempts by Pesce (5) to synthesize the mineral were unsuccessful and his work was confirmed by us.

The reported physical properties of "arizonite" were duplicated when ilmenite sand was heated overnight at 900°C . The treatment destroyed the ilmenite structure and the x -ray pattern was quite poor. However, if the sand was ground to 200 mesh and oxidized, the x -ray pattern of Fe_2TiO_5 and rutile was obtained, confirming Ramdohr's observation (8). The end products depend upon the temperature and at lower temperatures Fe_2O_3 and anatase are to be expected.

Prints of the only extant specimen (6) of Arizonite (U. S. National Museum Collection No. 86,973) together with hematite, and ilmenite taken by W. E. Richmond and measurements made on the original films by J. M. Axelrod were obtained through the courtesy of Dr. W. T. Schaller of the U. S. Geological Survey. Mr. Richmond's conclusion was that "the specimen labeled arizonite gives a powder photograph nearly identical with that of hematite. However, there are sufficient differences in the spacings and arrangements of the diffractive lines to preclude its identity with hematite. The powder photographs of hematite, arizonite, and ilmenite are all similar but the spacings of the same diffracting planes of each mineral are different. This probably means that all three minerals have the same structure cell but the cell edge lengths vary slightly with composition." Dr. Schaller also said that beach sands reported to be arizonite gave no x -ray pattern or a very poor pattern. Our examination (Table 1) of the print and the spacing measurements showed that arizonite is an impure mixture of hematite, ilmenite, anatase and rutile. All of the lines in the pattern can be accounted for by these compounds. This is sufficient evidence to discredit the mineral.†

In our opinion "arizonite" is weathered ilmenite, contrary to the opinions of Koenigsburger (2) and Moore (3). Purified ilmenite gained weight on heating at 360° , 600° and 800°C . and extrapolation by meth-

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† We are indebted to Mr. Axelrod who re-examined the x -ray evidence recently and concluded that arizonite is a mixture, principally anatase with hematite doubtfully present (spacings correct, but intensities not checking); and a fine-grained phase present, whose pattern is very similar to that of ilmenite but with spacings different from those of natural ilmenite which he has studied.

ods used by Pilling and Bedworth (7) indicated that ilmenite oxidation could be expected at 100° C. in months and at room temperature in years. Geologically, this is a short time. The occurrence of "arizonite" in tropical beach sands which are exposed to strongly oxidizing conditions and the absence of "arizonite" in massive deposits is in accordance with our hypothesis.

A convenient method for observing alteration of ilmenite by oxidation is to mount the sand in lucite resin, polish and examine the specimen under vertical polarized light. Unaltered ilmenite is black, oxidized ilmenite is red or brown and rutile inclusions are bright. These differences can be recorded by red sensitive film.

TABLE 1. COMPARISON OF ARIZONITE SPACINGS WITH THOSE OF HEMATITE, ILMENITE, RUTILE AND ANATASE

	Arizonite ¹		Hematite ¹		Ilmenite ¹		Rutile ²		Anatase ²	
	<i>d</i> (A.U.)	I/I ₀	<i>d</i> (A.U.)	I/I ₀	<i>d</i> (A.U.)	I/I ₀	<i>d</i> (A.U.)	I/I ₀	<i>d</i> (A.U.)	I/I ₀
<i>β</i> of 3.50	3.87	0.3	3.67	0.6	3.70	0.3	3.24	0.8	3.52	1.0
	3.67	0.4								
	3.50	1.0								
	3.23	0.3								
<i>β</i> of 2.66	2.96	0.1	2.68	1.0	2.73	0.1	2.49	0.6	2.37	0.24
	2.66	0.9								
	2.51	0.8								
	2.48	0.0								
<i>β</i> of 2.196	2.419	0.05	2.196	0.4	2.218	0.3	2.19	0.30	1.88	0.40
	2.370	0.1								
	2.196	0.2								
	2.180	0.0								
	2.080	0.0	3	1.885	0.1	1.854 ^b	0.4	1.70	0.28	
	1.885	0.1								
	1.863	0.0								
	1.833	0.2								
	b 1.687	0.7	1.690	0.2	1.716	0.8	1.69	1.0	1.66	0.24
	1.660	0.3								
	1.624	0.05								
	1.595	0.1								
	b 1.478	0.3	1.595	0.3	1.498	0.6	1.485	0.20	1.480	0.24
	1.449	0.2								
	1.451	0.5								
	1.462	0.6								
	1.359	0.1	1.451	0.5	1.462	0.6	1.449	0.20	1.362	0.08
	1.334	0.1								
	1.308	0.0								
	1.335	0.3								
	1.308	0.0	1.308	0.2	1.335	0.3	1.355	0.30	1.335	0.08
	1.261	0.2								
	1.267	0.2								
	1.262	0.11								
	1.162	0.05	1.161	0.2	1.267	0.2	1.091	0.08	1.164	0.08
	1.137	0.1								
	1.101	0.05								
	1.100									
1.088	0.05	1.0525		1.048	0.0	1.040	0.08	1.045	0.03	
1.052	0.05									
1.042	0.05									
		1.100		1.048	0.0	1.040	0.08	1.045	0.03	

¹ Spacings measured by U. S. G. S.

² Data from Hanawalt card index.

³ Hanawalt index gives $d=2.07$, $I/I_0=0.2$, obtained by one observer.

⁴ Hanawalt index gives $d=1.137$, $I/I_0=0.5$, not obtained by all observers.

⁵ Hanawalt index gives $d=1.86$, $I/I_0=0.6$.

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THE UNIT CELL OF MALACHITE

LEWIS S. RAMSDELL¹ AND C. WROE WOLFE²

The only unit cell data on record for malachite are those of Brasseur (1932). The authors of this paper, each working independently, found that there is a considerable error in the value of the angle β , as reported by Brasseur.

The axial ratio given in Dana (1909) for malachite is that of Lang (1863-4), who reported the values 0.8809:1:0.4012, with $\beta = 61^\circ 50'$. In this orientation the prominent cleavage is $\{001\}$. Goldschmidt (1918) chose a setting with the cleavage as $\{101\}$, and gave the axial ratio as 0.7823:1:0.4036, with $\beta = 91^\circ 03'$.

In his work on the crystal structure of malachite, Brasseur assumed the Goldschmidt value of $91^\circ 03'$ for β . The unit cell dimensions reported by him are $a_0 = 9.38kX$, $b_0 = 11.95$, $c_0 = 3.18$, giving an axial ratio of

MALACHITE. Angle Table*

$$a:b:c = 0.7914:1:0.2691; \beta = 98^\circ 44'; p_0:q_0:r_0 = 0.3400:0.2660:1$$

$$r_2:p_2:q_2 = 3.7598:1.2784:1; \mu = 81^\circ 16'; p_0' = 0.3440; q_0' = 0.2691; x_0' = 0.1536$$

Common forms	ϕ	ρ	ϕ_2	ρ_2	C	A
<i>c</i> 001	90°00'	8°44'	81°16'	90°00'	0°00'	81°16'
<i>b</i> 010	0 00	90 00	—	0 00	90 00	90 00
<i>a</i> 100	90 00	90 00	0 00	90 00	81 16	0 00
<i>m</i> 110	51 58	90 00	0 00	51 58	83 08	38 02
<i>p</i> 201	— 90 00	28 07	118 07	90 00	36 51	118 07

Less common forms: $x \bar{1}02$, $\gamma 011$, $\epsilon 111$.

Rare or uncertain forms: 130, 104, 101, $\bar{1}08$, 708, 134, 232, 562, $\bar{1}68$, $\bar{1}98$, $\bar{1}65$, $8 \cdot 15 \cdot 16$, $\bar{1}22$.

* Based on angles of Lang, but with the Ramsdell-Wolfe unit and orientation.

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0.7845:1:0.266. This value for c is just two-thirds of that given by Goldschmidt. Brasseur's values for b_0 , c_0 and d_{100} are closely checked by the authors, using the Weissenberg method, but they obtain a value of $98^\circ 42'$ for the angle β .

The relation between the unit cell thus found and the Goldschmidt unit is shown in Fig. 1. The $\{101\}$ cleavage of Goldschmidt becomes $\{201\}$ in the unit cell, and the value of c is reduced by one-third. It is not clear how Brasseur indexed his rotation films on the basis of $\beta = 91^\circ 03'$. The direction he used as $[100]$ is actually $[201]$ in the unit cell. No attempt to determine the structure of malachite has been made by the authors. But in view of the use of an erroneous value of β , the structure proposed by Brasseur must be regarded as questionable. The discrepancy between the morphological lattice and the x -ray lattice of Brasseur is, of course, explained by his improper value of β .

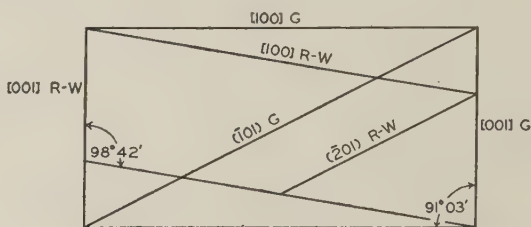


FIG. 1. Diagram showing relationship of the Goldschmidt (G) orientation of the morphological unit and the Ramsdell-Wolfe (R-W) structural unit cell.

Unit cell data

$a_0 = 9.49 \text{ \AA}$, $b_0 = 12.00 \text{ \AA}$, $c_0 = 3.24 \text{ \AA}$; $\beta = 98^\circ 42'$; $a_0:b_0:c_0 = 0.790:1:0.270$.

Space group $P2_1/a$.

Morphology

Previous morphological descriptions are transformed to the x -ray unit and orientation as follows:

Goldschmidt to Ramsdell-Wolfe: $\bar{1}0\frac{1}{3}/0\bar{1}0/00\frac{2}{3}$. The old elements thus transformed become $a:b:c = 0.7914:1:0.2691$; $\beta = 98^\circ 44'$. Dana to Ramsdell-Wolfe: $\bar{1}0\frac{4}{3}/0\bar{1}0/00\frac{2}{3}$.

Twinning

Twin plane $\{100\}$ is very common, often as penetration twins, sometimes polysynthetic. Twin index 1, obliquity $1^\circ 03'$. Complementary law twinning about $[201]$ also, as indicated by some x -ray photographs and by two measured crystals.

Physical properties

Cleavage $\{201\}$, which, considering the space group, is a plane of maxi-

imum spacing. Measured gravity on crystals 4.05 ± 0.02 . Calculated gravity 4.004.

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ANNOUNCEMENT OF NEW AWARD OF THE
MINERALOGICAL SOCIETY OF AMERICA

At the Council meeting of the Mineralogical Society of America held on November 10, 1949 it was voted to establish a new award to be given under the following conditions:

1. The name of the award shall be the Mineralogical Society of America Award.*
2. The award shall be life membership in the *Mineralogical Society of America*. A certificate as evidence of the award, signed by the President and Secretary of the Society, shall be presented to the recipient at the annual meeting.
3. The award shall be given to an individual for an outstanding contribution within the fields of interest of the Society. The contribution must be the published results of original research made during the three years prior to selection by the Council.
4. The work for which the award is given must have been accomplished at an age of thirty-five years or less.
5. The award shall be made without regard to nationality. Membership in the Mineralogical Society of America is not a necessary prerequisite.
6. The President shall appoint a committee to nominate a recipient. The selection, however, shall be subject to the approval of the Council.
7. The award shall be given annually only if a suitable candidate can be selected by the Council.

The committee appointed by President Tunell to select a recipient for this new award is:

N. L. Bowen, Chairman
J. E. Hawley
M. N. Short

The committee will welcome suggestions from Fellows of suitable candidates for the award.

C. S. HURLBUT, JR., *Secretary*

* This name is to be used temporarily until a more suitable name is found.

PROCEEDINGS OF SOCIETIES

THE CRYSTALLOGRAPHIC SOCIETY OF AMERICA

WILLIAM PARRISH, *Secretary-Treasurer, c/o Philips Laboratories, Inc.,
Irvington-on-Hudson, New York.*

The fourth annual Spring meeting was held at University of Michigan, Ann Arbor, Michigan, April 7-9, 1949. 97 members and visitors registered for the meeting.

The officers for 1949 were:

President: Professor J. D. H. Donnay, The Johns Hopkins University.

Vice-President: Mr. Samuel G. Gordon, The Academy of Natural Sciences of Philadelphia.

Secretary-Treasurer: Dr. William Parrish, Philips Laboratories, Inc.

Councilors: Professor Raymond Pepinsky, Pennsylvania State College. Professor George Tunell, University of California at Los Angeles.

After more than a year's discussion the Society voted to join with American Society for X-ray and Electron Diffraction to form a single new Society, the *American Crystallographic Association*, which will carry on the functions of the two previous Societies. Information on the new Society may be obtained from the Secretary, Dr. Howard T. Evans, Jr., c/o Philips Laboratories, Inc., Irvington-on-Hudson, N.Y.

Abstracts of papers given at the technical sessions are given below in order of presentation.

CRYSTAL STRUCTURE

Chairmen: I. Fankuchen, Polytechnic Institute of Brooklyn,
A. Pabst, University of California, Berkeley, California.

CRYSTALLOGRAPHIC SYMMETRY IN RECIPROCAL SPACE AND IN VECTOR SPACE

M. J. BUEGER, *Massachusetts Institute of Technology.*

The transformation of objects into reciprocal space and into vector space is important in crystal structure studies, so it is not only of theoretical interest, but of some practical importance to see how crystallographic symmetry is revealed in these spaces.

Application of the origin-shift theorem shows that a symmetry operation through the origin, and having translation component of c/q , appears as the same symmetry operation in reciprocal space, except that it causes a phase shift between fields of $e^{2\pi il/q}$. It follows that the fields related by the several operations of a symmetry element through the origin have phases related by $e^{2\pi ipl/q}$, where p is the power of the operation relating the fields. If the symmetry element occurs displaced from the origin by $xa+yb$, then the phase of the point is further shifted by $e^{2\pi i(xh+yk)}$. Hence the symmetry of reciprocal space is the same as the point-group symmetry of direct space, except that the phases of the fields are shifted in accordance with the discussion above. Obviously each space group has a unique reciprocal representation consisting of point-groups symmetry, reciprocal lattice point locations, and phase fields.

The transformation from crystal to vector space involves the transfer of each image-point of a symmetrical set to the origin of vector space. Furthermore, the periodicity of vector space is identical with that of crystal space. From this it follows that to each space group in crystal space the corresponding symmetry in vector space is the arithmetic space group. The translation components of the symmetry elements in crystal space appear in vector space as a collection of point images removed by that translation from the origin. Taking into account the centrosymmetrical aspect of vector space, it follows that all space groups in crystal space can be distinguished in vector space except those particular pairs which are differentiated by the operation $\bar{1}$ only, such as $R3$ and $R\bar{3}$. This result corresponds with the implication-theory result given at the Lake George meeting of the *A.S.X.R.E.D.* in June, 1946.

ON THE DERIVATION OF HARKER-KASPER INEQUALITIES*

CAROLINE H. MACGILLAVRY, *Alabama Polytechnic Institute* (temporarily).

The derivation given by Harker and Kasper of the inequalities relations between structure factors¹ involves quite laborious calculations for others than the simplest space groups and does not show clearly the fundamental meaning of the constant term, which is evidently $1/n$ where n is the symmetry number of the space group. It was shown that the fundamental Harker-Kasper inequalities can be easily found by straightforward group-theoretical methods² for any space group.

Moreover, it was pointed out that Harker-Kasper inequalities hold only if the electron density is a positive function; that is, if the scattering can be considered as Thomson-scattering from free electrons.

* This work was partly supported by the Office of Naval Research, Contract No. N7onr-377 with the Auburn Research Foundation.

¹ Harker, D., and Kasper J. S., *Acta Cryst.*, **1**, 70 (1948).

² See for example Zachariasen, W. H., *Theory of X-Ray Diffraction in Crystals*, John Wiley, 1945

THE PHASES AND MAGNITUDES OF THE STRUCTURE FACTORS

J. KARLE AND H. HAUPTMAN, *U. S. Naval Research Laboratory, Washington, D. C.*

A fundamental set of inequalities exists among the Fourier coefficients which represent a positive function. It is found that if the first n coefficients are given, the $n+1$ coefficient must be restricted to within narrow bounds if a positive function is to be represented. This idea finds application in the determination of the structure of crystals since the electron density function is a positive function whose Fourier coefficients are the structure factors. The investigation considered here is the generalization of the pioneer work of Harker and Kasper.¹ The relationships of Harker and Kasper follow from our derivation. In addition, we obtain infinitely more inequalities.

It is not only possible to determine the signs of real coefficients but it is also possible to pin down the values of the real and imaginary parts of complex coefficients. By using the relationships which are necessary and sufficient, it is possible to derive coefficients beyond the range in which their magnitudes may be experimentally determined.

¹ Harker, D., and Kasper, J. S., *Acta Cryst.*, **1**, 70 (1948).

ONE-DIMENSIONAL REPRESENTATION OF TRIPERIODIC FUNCTIONS

J. D. H. DONNAY, *The Johns Hopkins University*,

AND

GABRIELLE E. HAMBURGER, *Massachusetts Institute of Technology*.

Any triperiodic function, say the electron density in a crystal, can be represented by a one-dimensional function. The latter is evaluated along the row $[n^2n1]$. Provided n is sufficiently large and the function is computed at enough points, evaluating the function along the period of the row approximates evaluating it throughout the cell. Let $n=1000$ and let the function be computed at any one of n^3 equidistant points within the period. Let X be the new x coordinate and H the new h index after transformation $n^2n1/0n1/001$. Since $H=n^2h+nk+l$, there is a unique H for every hkl . We may therefore replace F_{hkl} by F_H and $\sum_h \sum_k \sum_l$ by \sum_H . The point on the row that most closely corresponds to a given xyz is $X_1=x'+y'/n+x/n^2$, where $y'=y$ if $x<0.5$ and $y'=y-0.001$ if $x\geq 0.5$, and likewise $z'=z$ if $y<0.5$ and $z'=z-0.001$ if $y\geq 0.5$. Conversely, to a given point $X(0\leq X<1)$ there corresponds an equivalent point with trimetric coordinates $x=n^2X$, $y=nX$, $z=X$ (owing

to periodicity, only the decimal parts of x and y are used). Hence $lx+ky+lz=(n^2l+nk+l)X=HX$. We may therefore write for the electron density at X ,

$$\rho(X) = (1/V) \sum_H F_H e^{-i1000HX},$$

where the angle is expressed in millicycles ($1mC=2\pi/1000$), as obtained directly from the calculations, in order to take advantage of the decimalization of the circumference (Villarcéau)¹ and of recently calculated tables.²

¹ Villarcéau, Yvon, *C. R. Ac. Sc. Paris*, **70**, 1233-1236; **71**, 362-368 (1870).

² Donnay, J. D. H., and Hamburger, G. E. (1948), Tables for Harmonic Synthesis giving the terms of Fourier series to one decimal at every millicycle tabulated for coefficients 1 to 100 and fiducial cosine values to eight decimals. Baltimore, Md. (1948).

OPERATION OF THE TWO-DIMENSIONAL FOURIER TRANSFORMER*

RAY PEPINSKY, *Alabama Polytechnic Institute*.

Computations of density functions and structure factors are illustrated, as carried out by our two-dimensional Fourier transformer.¹ The sources of error in the instrument are analyzed, and procedures for minimizing these described. A discussion is given of operational test procedures; of methods for addition, subtraction, multiplication and differentiation of functions; of simultaneous storage of density and scattering functions in the machine, when the density function is centrosymmetric; of the method for extension of range of coefficients; and of application of the machine in special analytical methods.

* This development has been supported by the Office of Naval Research, under Contract N7onr-377 with the Auburn Research Foundation.

¹ Pepinsky, R., *Jour. Appl. Phys.*, **18**, 601 (1947).

PATTERSON-HARKER MAPS OF PROTEIN CRYSTALS

DOROTHY WRINCH, *Smith College*.

In using 'experimental' Patterson-Harker maps of protein crystals to test any theory of protein structure, the question arises as to the relation between these maps of *crystals* comprising molecules in a water medium and maps of the *molecules* alone.

(1) Since the vector maps of the molecules+medium and of the molecules differ by the interactions of the medium with itself and with the molecules, we cannot derive one from the other without knowing the structure of the molecules.

(2) However, if 2 crystals in S_1 differ by a constant at every point, their S_2 vector maps, differing by the interactions of a *constant* distribution with itself and with another distribution, also have this property. Hence by suitably reducing every entry in a P-H map, we obtain the S_2 map of the 'reduced' crystal with a zero intermolecular density, thereby replacing the molecules by 'reduced' molecules in which the true electron density is replaced by its deviation from the intermolecular density.

(3) To test any 'experimental' P-H map against a proposed structure, we therefore require the vector map, not of the structure, but of the structure reduced to the appropriate level.

In testing the cyclol theory of protein structure against published P-H maps of proteins, we make, at this stage, the simplifying assumption of constant densities outside and inside the molecular cage skeletons. In a calculation involving over 150,000 terms, vector maps have been obtained for point atom distributions representing the C_1 cage and its interior reduced to a series of levels. The resulting maps show a highly characteristic morphology, their step by step derivation permitting us to diagnose the features in S_1 which are responsible.

The maps are compared with published 'experimental' P-H maps of protein crystals.

THE STRUCTURE OF AMMONIUM DIHYDROGEN PHOSPHATE, $\text{NH}_4\text{H}_2\text{PO}_4$

B. CHALMERS FRAZER, *Alabama Polytechnic Institute.*

The structure of ammonium dihydrogen phosphate¹ was determined by us in September, 1948. Some time later we learned² that R. Uedo³ had published the structure in May, 1948. The results of the two investigations agree fairly well crystallographically, and are in conclusive agreement on the one point of real interest, namely, the existence of an $\text{N-H} \cdots \text{O}$ as well as an $\text{O-H} \cdots \text{O}$ system of hydrogen bonds in the structure. This should be of considerable importance in accounting for the electrical peculiarities of this crystal.

The results of the two structure determinations are discussed and compared with the well-known KH_2PO_4 structure.⁴ Principal attention is centered on the hydrogen bond system and the possible hydrogen configurations as related to the normal and the polarized state. Speculations concerning the normal and polarized states are made from the viewpoint of a transition from *I42d* to *Fdd*.

¹ Frazer, B. C., *Stroboscopic X-ray Diffraction Investigation of Piezoelectrically Oscillating Crystals*, Auburn Research Foundation, Tenth Quarterly Report to the U. S. Army Signal Corps, Contract No. W 36-039 sc-32031, 15 September, 1948.

² Private communication from A. L. Patterson.

³ Uedo, R., *X-Rays (Japan)* **5**, 21 (1948).

⁴ West, J., *Zeit. Krist.*, **74**, 306 (1930).

THE CRYSTAL STRUCTURE OF SiC , TYPE 10-H

L. S. RAMSDELL AND J. A. KOHN, *University of Michigan.*

A crystal of SiC , which did not correspond to any of the known types on the basis of crystal measurements, was shown by Weissenberg photographs to have a 10-layer hexagonal structure, and is accordingly designated type 10-H. Such a structure does not fit into any of the previously suggested series, and therefore no clues were available for indicating which of the various geometrical possibilities might be involved. Intensity calculations were carried out for eight different possible structures. The only one in harmony with the observed intensities is designated by the symbol 3223. This represents the zig-zag sequence of the Si (or C) atoms along the three symmetry axes in the 1120 plane.

This arrangement has the symmetry $C3m$, which should appear on the films as $C\bar{3}m$. However, both the films and the calculated intensities show 6-fold symmetry. The explanation of this apparently higher symmetry is given.

THE PROBLEM OF THE GRAPHITE STRUCTURE

JOSEPH S. LUKESH, *Knolls Atomic Power Laboratory,* General Electric Company,*
AND

LINUS PAULING, *California Institute of Technology.*

Certain experimental observations cannot be explained readily if the structure of graphite is as generally accepted. A new structure is suggested having the following constants: $a_0 = 2.456 \text{ \AA}$, $b_0 = 4.254$, $c_0 = 6.696$. Space-group: $D_{2h}^{23} - Fmmm$. There are eight atoms in the cell at O, $\pm U$, O, where U is nominally one-sixth.

Observations of angular relations of individuals indicate that the C-C bonds are not all equal. This is supported by structure factor computations and by theoretical considerations of bond numbers in resonance systems. Further work is necessary before the suggested structure can be unequivocally accepted.

* The work reported here was carried out for the Atomic Energy Commission under contract No. W-31-109 Eng-52.

A STRUCTURAL CLASSIFICATION OF THE ALUMINO-FLUORIDES

Address of the Retiring President of the Crystallographic Society of America

A. PABST, *University of California.*(To be printed in full in *The American Mineralogist*)

SYMPOSIUM ON TWINNING

Chairmen: J. D. H. DONNAY, *The Johns Hopkins University.*M. J. BUEGER, *Massachusetts Institute of Technology.*

THE NEW APPROACH TO THE STUDY OF TWINNING

J. D. H. DONNAY, *The Johns Hopkins University.*

THE DETERMINATION OF THE TWIN LAWS IN CRYSTAL PROJECTIONS

M. A. PEACOCK, *University of Toronto.*

THE GENESIS AND STRUCTURE OF TWINS

M. J. BUEGER, *Massachusetts Institute of Technology.*

THE GENESIS OF GROWTH TWINS

C. FRONDEL, *Harvard University.*

THE ORIGIN OF ANNEALING TWINS

ROBERT MADDIN, *Yale University.*

The slip process in face-centered metals is never as simple as the conventional Taylor and Elam analysis, for there is always a second cooperating plane which contains the same slip direction as the primary plane. There is also another cooperating plane which is commonly called the conjugate slip plane, previously observed to function only when the rotation due to the action of the first plane brought about a symmetrical condition producing equal shear stresses on the two systems.

Annealing twins in face-centered metals could arise from preformed nuclei which could be described as twin faults produced in a two-stage slip process. Such a process, which has been postulated by C. H. Mathewson for many years, is one in which the $\{111\}$ planes can move stepwise in two adjacent $\langle 112 \rangle$ directions integrating into the $\langle 110 \rangle$ slip direction. In the present investigation carefully tapered single crystals were strained and annealed at a high temperature, and twins were found with their composition planes parallel to the active slip planes and with no others observable. This can be interpreted as a very strong evidence in favor of the two-stage slip process.

X-ray study of the strained single crystals exhibited a sharp rise in the shear-strengthening curves at a value of shear of about 0.22. Recrystallization was found to begin at this value of shear. The orientations of ten of the new grains, determined by x-rays, were related to the parent lattice by a rotation about lines perpendicular to the three acting slip planes.

EFFECTIVE LATTICE ROTATIONS PERMITTED BY SHORT
DISPLACEMENTS OF LATTICE POINTSM. L. KRONBERG AND F. H. WILSON, *American Brass Company.*

If a lattice is rotated parallel to one of its nets about a lattice point in the net, there is a series of angular positions in each of which a definite proportion of the lattice points will

coincide with points of the lattice before rotation. These points define a multiple of the primitive lattice. The relationship between a pair of orientations, consisting of the original and any one of the series, can also be considered as a 180° rotation about a line in the net through the point of rotation and the nearest coincidence site. The relationship is developed in detail for the specific example of a 38° rotation of a face-centered cubic lattice about a $[111]$ axis. This development shows that because of the periodicity of coincidence sites, one orientation can convert to the other by relatively simple displacements of the remaining lattice sites. The existence of this coincidence mechanism was found during an attempt to understand the experimental observation that copper can give rise to a recrystallization orientation that is related to the parent orientation by a 38° rotation about a $[111]$ axis. Other similar examples have been observed in metals.

TWINNING IN NESQUEHONITE, $\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$

MAY RISCH KINSOLVING, CAROLINE MACGILLAVRY AND RAY PEPINSKY,
Alabama Polytechnic Institute.

It was shown by one of us¹ that nesquehonite, described as orthorhombic in the literature, has actually a lower symmetry; the pseudo-orthorhombic symmetry is caused by repeated twinning, as shown by close doubling of many maxima on oscillation diagrams. From Weissenberg diagrams it has now become possible to determine the true unit cell and the twin law. The crystals are monoclinic, the ortho-axis being identical with that formerly called the c -axis. If we take the axes corresponding to the originally assigned orthorhombic cell, the space group is found to be $P2_1/n-C_{2h}^2$; $a = 7.68 \text{ \AA}$, $b = 5.39 \text{ \AA}$ (formerly called c), $c = 12.00 \text{ \AA}$ (formerly called b), $\beta = 90.45^\circ$. The angle β was measured accurately on Weissenberg diagrams in which a 9 cm. shift of the film corresponded to a rotation of 12.7° of the crystal. The twinning occurs on the monoclinic (001) plane. A complete structure determination is now in progress.

¹ Pepinsky, R., *Phys. Rev.*, **59**, 925 (1941).

TWINNING IN MICROCLINE

FRITZ LAVES, *University of Chicago.*

Some observations on twinned microcline indicate that four different twin positions are related to each other so as to produce one set of planes in reciprocal space. X -ray photographs show that the four spots corresponding to planes with the same hkl values are joined by faint streaks. The probable explanation of this effect is discussed.

CRYSTAL GROWTH

Chairman: J. W. GRUNER, University of Minnesota.

OBSERVATIONS ON THE FLAME FUSION GROWTH AND STRUCTURE OF RUTILE CRYSTALS

CHARLES H. MOORE, L. MERKER AND L. E. LYND,
Titanium Division, National Lead Co.

In the growth of single crystals of rutile by the present process, certain fundamental differences are noted from the synthesis of corundum or spinel by the Verneuil process. The tendency of TiO_2 to lose oxygen near its melting point necessitates that the crystal be grown in an oxygen-rich atmosphere. Even under these conditions, the resulting crystal is black in color, although apparently identical with the colorless material, judging from unit-

cell measurements and x-ray powder patterns. Upon subsequent oxidation the crystal can be made substantially colorless.

Variations from stoichiometric proportions of constituents are permissible with rutile, indicating a structural flexibility not heretofore emphasized. A discussion of the mechanism of these variations is given, and differences in physical and electrical properties with degree of oxidation are described. The technique and theory of growth is discussed, as well as the possibility of extending the same principles to the growth of oxides of other members of the atomic transition series.

THE FLAME FUSION SYNTHESIS OF MULLITE

W. H. BAUER, I. GORDON AND C. H. MOORE, *Rutgers University.*

In conjunction with the O.N.R. project on tourmaline synthesis, a single crystal of an incongruently melting silicate, mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$), has been grown by the flame fusion process. The equipment consists of a feeding device, burner, lowering apparatus and suitable gas controls. A modified Verneuil type burner employing hydrogen and oxygen was used. A feed of extreme purity and low density was developed by calcining a stoichiometric mixture of alum [$\text{Al}_2(\text{SO}_4)_3 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$] and pure silica gel.

X-ray and optical data show the resulting boule to be a single crystal of mullite. The d/n values obtained all correspond to those reported in the *A.S.T.M.* cards and no evidence of the existence of any crystalline alumina or silica was noted. The largest single crystal grown was 2 cm. in length and 1 cm. at its thickest point.

This synthesis presents a new approach to the field of silicate research. It indicates that the flame fusion technique is feasible for the study and possible single crystal growth of certain anhydrous silicates, provided that such problems as feed preparation, viscosity of the molten surface, and possible polymorphous inversions can be successfully solved or circumvented.

THE MUTUAL MODIFICATION OF THE CRYSTAL HABIT OF AMINO ACIDS AND SODIUM CHLORIDE

CHARLES P. FENIMORE, *The Johns Hopkins University*

AND

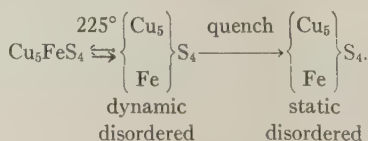
ARTHUR THRAILKILL, *Aberdeen Proving Ground.*

Glycine, pyridine betaine, and β alanine in aqueous sodium chloride solutions modify the crystal habit of growing sodium chloride: the first causes the formation of dodecahedra, the other two give octahedra. Glycine and β alanine are themselves modified by sodium chloride. The largest or most unexpected (on the basis of the modified law of Bravais) new form developed on the amino acid by sodium chloride is compared with the form developed on sodium chloride by the amino acid. A fairly good correspondence of mesh dimensions is found in both cases and a structural correspondence is found in the case of glycine.

DISORDER IN BORNITE, Cu_5FeS_4

ALFRED J. FRUEH, JR., *Massachusetts Institute of Technology.*

Diffraction data from single crystals of bornite, from Bristol, Connecticut, indicate that bornite in its ordered form is tetragonal with cell dimensions: $a = 21.91 \text{ \AA}$, $c = 10.95 \text{ \AA}$. When quenched from above 225° C. the crystals become isometric, $a = 10.95 \text{ \AA}$. This can be explained by disordering between the copper and iron,



The absorption of heat that accompanies the disordering has been observed by means of a thermal analyzer.

The appearance of super structure lines and their consequent disappearance upon heating has been observed in powder patterns of bornite from various localities, both statically and dynamically, with the aid of a high temperature camera. It has been found possible to reorder the disordered material by slow cooling from 225° C.

CRYSTAL OPTICS

Chairman: A. N. WINCHELL, University of Virginia.

THE FREQUENCY-DISTRIBUTION OF THE OPTICAL PROPERTIES OF CRYSTALS

A. F. KIRKPATRICK, American Cyanamid Company.

A study has been made of the frequency-distribution of approximately 1000 crystalline phases, both inorganic and organic, among the following classes: isotropic, uniaxial positive, uniaxial negative, biaxial positive, and biaxial negative. The distribution of the refractive indices, N , N_O , N_E , N_X , N_Y , N_Z , of specific gravity, and of oblique and parallel extinction has also been examined within the above classes.

The relationships of the results to the examination of crystalline substances in an industrial analytical laboratory are discussed. For example, a narrower interval in refractive index between individual standard immersion liquids is needed near the index with the greatest frequency of occurrence than at index ranges of low frequency. Emphasis is placed on the relations of the distributions to the certainty of the identification of an unknown phase by the determination of its optical properties.

CRYSTAL OPTICS ON MICROSCOPIC VIEWS—A MONOCLINIC CASE

W. A. O'BRIEN, Celanese Corporation of America,

AND

J. D. H. DONNAY, The Johns Hopkins University.

In the microscopical study of crystals, measurements of refractive indices and determinations of fast and slow extinction directions may be made on various faces. While the morphology of the crystal can be determined by microscope goniometry¹ it is also desirable to derive the principal refractive indices and the optical orientation from the measured optical properties. The procedure will vary with the nature of the data. Graphical methods are presented for the transformation, when the available data are the fast and slow extinction directions in two faces, the refractive indices in one of the two faces, and the orientation of the two faces with respect to each other and to a plane of optical symmetry. (The faces must not be perpendicular to a plane of optical symmetry.) This case arises in monoclinic crystals. The transformation of the measured refractive indices into principal refractive indices is based on Mertie's monogram² used for the solution of the ellipse from angular data determined from the stereographic projection. The determination of the optical orientation is based on finding the locus of possible optic axis positions for each face and noting the intersection of the loci for the two faces. This is done by stereographic projection methods.

¹ Donnay, J. D. H., and O'Brien, W. A., *Ind. and Eng. Chem., Analyt. Ed.*, **17**, 593-597 (1945).

² Mertie, John B., *Am. Mineral.*, **27**, 538-551 (1942).

SOME PHOTOELASTIC CONSTANTS OF CRYSTALS $\text{NH}_4\text{H}_2\text{PO}_4(\text{ADP})$ AND $\text{KH}_2\text{PO}_4(\text{KDP})$

C. D. WEST AND A. S. MAKAS, *Polaroid Corporation.*

The results of our static measurements for these crystals, which belong to the class V_d-42m , are piezoelectric, and were supplied by the Brush Development Company, are found in the accompanying table. We also confirmed experimentally the correctness of Pockels' theory (1906) for tetragonal crystals, no examples of which were previously studied.

Line	Quantity	ADP	KDP
1	ω_D, ϵ_D	1.5246, 1.4792	1.5095, 1.4684
2	C_1, C_3	$-1.84 \pm .17, 32.2 \pm .7$	$(>0, <0.5), 19.4 \pm .4$
3	$2(S_{11}-S_{12}), S_{66}^E$	20, 164	43, 164
4	$(q_{11}-q_{12}), q_{66}^E$	1.04, -18.15	$(>-0.3, <0), -11.25$
5	$\frac{1}{2}(c_{11}-c_{12}), c_{66}^E$	50, 6.1	24.2, 6.1
6	$\frac{1}{2}(p_{11}-p_{12}), p_{66}^E$	0.052, -0.1107	$(>-0.007, <0), -0.0685$

Line 1 shows refraction indices after Topsoe and Christiansen (1873), lines 3 and 5 elastic shear compliance resp. stiffness after Mason (1946), lines 2, 4 and 6 are various expressions of photoelastic shear constants in the notation of Szivessy (1928). The wavelength $0.560 \text{ m}\mu$ applies to line 2. For lines 2, 3 and 4 the units are $10^{-13} \text{ cm}^2/\text{dyne}$, for line 5 they are $10^{10} \text{ dyne/cm}^2$.

INFRARED MICROSCOPY

E. L. PERRINE AND W. C. McCRONE, *Armour Research Foundation of
 Illinois Institute of Technology.*

Plans and details of construction for a very simple infrared microscope are shown and discussed. The unit involves essentially a high voltage transformer and a secondary photo-emission tube (1-P-25) similar to those used during the war in the sniperscope. The instrument has been constructed to fit into any microscope in place of the ocular.

The applications of this instrument to crystallography and microscopy are briefly discussed. These include the examination of materials opaque to visible light (some of the minerals, organic dyes, etc.) and the study of crystal optics. The present limitations and possible improvements for the infrared microscope are considered.

AN INEXPENSIVE PETROGRAPHIC MICROSCOPE

HORACE WINCHELL, *Yale University.*

A number of manufacturers have recently announced development of students' microscopes selling for about \$100.00, which can be adapted for elementary petrographic work by the addition of inexpensive polaroid elements, and a simple rotating stage. The design of a stage and installation of polaroids are described.

MISCELLANEOUS PAPERS

Chairman: E. H. KRAUS, University of Michigan.

**ELECTRICAL EVIDENCE ON THE CLASS OF
SOME TRICLINIC CRYSTALS**

W. L. BOND, Bell Telephone Laboratories.

An example of a crystal class should have two qualifications: there should be no doubt that it belongs to the class it "represents," and it should be readily available for study. It should not be unstable, as are the "examples" of class 1 quoted in crystallography texts. The "classical example" used in most mineralogy texts and in Bunn is calcium thiosulfate hexahydrate—it dehydrates in a day or so and probably belongs in class $\bar{1}$ since it is not piezoelectric and most crystals are of pinacoidal habit. Rubidium ferrocyanide dihydrate (Phillip's example) is stable, but is neither pyroelectric nor piezoelectric, and hence is probably of class $\bar{1}$. Strontium ditartrate tetrahydrate (Cady's example) is both pyroelectric and piezoelectric but extremely unstable, dehydrating violently in minutes. Phillips also says that axinite probably belongs to class 1, but since we find it to be neither pyroelectric nor piezoelectric, we think it belongs to class $\bar{1}$.

The liquid air tests for pyroelectricity are open to criticism as not testing at room temperature—conceivably a crystal could transform to a pyroelectric class before reaching liquid air temperature, then give a positive test. Piezoelectric tests are not open to this criticism.

Triclinic crystals containing an optically active organic acid are certainly of class 1 since the optically active component cannot have a center of symmetry. One developed at the Bell Telephone Laboratories is aminoethyl ethanolamine hydrogen tartrate. It is pyroelectric, piezoelectric and stable.

MAXIMUM HARDNESS VECTORS IN THE DIAMOND

C. B. SLAWSON AND J. A. KOHN, University of Michigan.

Since, in the lapping of diamonds, it is desirable to work on the so-called "on-grain" or softer lapping directions, little information is available concerning other hardness vectors. Qualitative determinations, by the use of standard diamond-lapping equipment, have been made on those "off-grain" lapping directions which parallel zonal lines connecting cube, octahedron, and dodecahedron faces.

When lapping parallel to a given direction, it is found that as one departs farther and farther from the "on-grain" position, a point is eventually reached where an abrupt variation in susceptibility to lapping occurs. These "limits" of "off-grain" lapping directions have been approximately evaluated and the results plotted on stereographic projections. All lapping directions investigated gave maximum hardness areas displaying three fold symmetry about the octahedron face and, likewise, two-fold symmetry about the dodecahedral position. Such areas beyond the "limits" offer the hardest and, therefore, most suitable directions for the working surfaces of diamond tools.

Several difficulties were encountered in the standardization of lapping conditions: (a) it is impossible to maintain a surface of uniform lapping efficiency over any extended period of time, (b) twinning lamellae were consistently present to a variable degree, (c) criteria for defining "limiting" conditions cannot be precisely established.

CRYSTALLOGRAPHIC DATA ON WAVELLITE FROM LLALLAGUA, BOLIVIA AND ON CACOXENITE FROM HELLERTOWN, PENNSYLVANIA

SAMUEL G. GORDON, *The Academy of Natural Sciences of Philadelphia.*

Wavellite, $[\text{Al}_3(\text{PO}_4)_2(\text{OH}, \text{F})_3 \cdot 5\text{H}_2\text{O}]_4$, *Pcnn*. The unit-cell dimensions are: $a_0 = 9.60 \text{ \AA}$, $b_0 = 17.31$, $c_0 = 6.98$; $a_0/b_0 = 0.555$, $c_0/b_0 = 0.403$, $c_0/a_0 = 0.727$. By goniometry, $a = 0.554$, $c = 0.403$, $p_0 = 0.726$. $M = 1652$ (from formula), 1649 (calculated from cell). Sp. gr. = 2.36 (on weighing), 2.365 (calc.).

Cacoxenite, $[\text{Fe}_4(\text{PO}_4)_3(\text{OH})_3 \cdot 12\text{H}_2\text{O}]_{12}$, *C6/mmm(?)*. The unit-cell dimensions are: $a_0 = 27.6 \text{ \AA}$, $c_0 = 10.4$; $c_0/a_0 = 0.377$. $M = 9312$ (from formula), 9340 (calculated from cell). Sp. gr. = 2.26 (on weighing), 2.25 (calc.). A new chemical analysis on 50 mg. gave: Fe_2O_3 , 40.5; P_2O_5 , 26.8; H_2O , 32.4 (= 99.7). (The lattice constants on cacoxenite were determined by Dr. Joseph Singer.)

WHAT IS A MINERAL?

A. N. WINCHELL, *University of Virginia.*

The old definition of a mineral as a natural inorganic substance of definite chemical composition is criticized. A new definition is proposed and its effects are illustrated.

A COMPACT DEMOUNTABLE X-RAY DIFFRACTION TUBE AND POWER SOURCE¹

HUGH MONTGOMERY LONG, JR., ANDREW SPEAR AND RAY PEPINSKY
Alabama Polytechnic Institute.

A compact diffraction unit has been constructed on the basis of earlier experience.² It contains a demountable x-ray tube with an electron gun to provide fine focus and grid control, a vacuum system, and stabilized high potential and current supplies. The tube body and electron gun are at ground level, the target being cooled by an oil circulating system to reduce leakage current to a few microamperes. The low average power demands of the unit permit the use of a stabilized radio-frequency high potential generator of the type used in electron microscopes. The table top is 33" high and of dimensions 34" × 42", and the unit is mounted on casters.

¹ Development supported by Signal Corps Engineering Laboratories, Contract W 36-039 sc-32031.

² Pepinsky, R., *Phys. Rev.*, **67**, 308 (1945); *Phys. Rev.*, **69**, 546 (1946); Long, H. M., and Pepinsky, R., *Phys. Rev.*, **74**, 126 (1948).

BOOK REVIEWS

THE STORY OF JADE. By the late Herbert P. Whitlock and Martin L. Ehrmann, pp. 222, 179 black illustrations and six full colored plates, 8"×11 $\frac{1}{4}$ ", Sheridan House, New York, 1949. Price \$12.50.

The Story of Jade is a very impressive volume. It contains a wealth of authoritative information concerning the occurrence and the various properties of jade, methods of carving, as well as the meanings of shapes and symbols of ancient jade. It has many beautiful illustrations in black and white and full colors. The book is well planned and excellently printed. It is a prize volume.

The authors are jade experts. The senior author, the late Herbert P. Whitlock was for twenty-three years curator of mineralogy at the American Museum of Natural History and after retirement in 1941, he became curator emeritus and research associate in jade. Mr. Whitlock was an ardent collector of jade and many of the illustrations are of objects from his extensive private collection. He spent many years in preparing the manuscript which was completed shortly before his death in 1948. Arrangements for the publication of this significant contribution on jade were made by the junior author, Mr. Martin L. Ehrmann, who has had wide experience as a dealer in minerals and is a competent connoisseur of jade.

The comprehensiveness of the text is indicated by the chapter headings, which are: Jade: The Many Colored Jewel of Heaven; The Jade of Ancient China; Dragons, Phoenixes and Other Creatures; Taoist Symbols; Gods and Immortals; How Buddhism Influenced Chinese Carved Jade; Salutations and Inscriptions; Bowls, Cups and Other Containers; Beads, Buckles and Other Articles of Adornment; and Various Objects Carved from Jade.

The very readable text is amplified by numerous illustrations of superbly carved objects from the Drummond, Cockcroft, Morgan, and Whitlock jade collections. The beautiful designs and the exquisite carvings reveal the marvelous artistry and skill of the Chinese worker in jade.

The Story of Jade is a "must" for all libraries. Mineralogists and mineral dealers, as well as collectors and lovers of gems, will find the book very stimulating and extremely helpful.

EDWARD H. KRAUS
University of Michigan

PROSPECTORS' MANUALS FOR RADIOACTIVE MINERAL DEPOSITS

Within the last two years, three governments have issued handbooks to aid the prospector in his search for deposits of radioactive materials. These guides are of interest to both amateur and professional geologists and mineralogists, for they contain not only summary descriptions of the chief radioactive minerals and the deposits in which they occur but also instructions for use of Geiger counters and other detection methods as well as statements of official prices and control regulations.

The Canadian booklet, *Prospectors' Guide for Uranium and Thorium Minerals in Canada*, was written by Hugh S. Spence and F. N. Senftle and is issued by the Bureau of Mines, Department of Mines and Resources, Ottawa, Canada, from whom it may be obtained (no cost is indicated). The chief topics discussed are uranium and thorium minerals, aids in identifying them, their mode of occurrence, and their determination by radioactive methods. Two appendices treat the use and care of the Geiger counter and examination of samples by the Department of Mines and Resources. A third presents an extract from the regulations affecting prospecting for radioactive materials in Canada.

The Department of Scientific and Industrial Research of the Geological Survey of

Great Britain has published *A Prospectors Handbook to Radioactive Mineral Deposits*, which may be obtained from the Department for sixpence (about 15 cents) at Rex House, 4-12 Regent Street, London, S.W.1. Because of the heavy British investment in atomic energy installations, the Ministry of Supply has guaranteed to purchase all high-grade uranium ore produced in the Colonial Empire during the next ten years at a minimum price of £1,540 per long ton of contained uranium oxide (13s. 9d per pound). Grants toward the construction of extraction plants and development of mines also may be made. The Atomic Energy Division of the Geological Survey is prepared to examine and assay samples of uranium ore.

After a series of descriptions of the more abundant radioactive minerals, the handbook summarizes the geology of various types of uranium and thorium deposits and lists world-wide examples. Next follow accounts of the main detection methods (Geiger counter, fluorescence, photography, etc.) and an announcement of the purchasing and aid program of the Ministry of Supply. A short glossary of pertinent geologic words and phrases concludes the work.

In the United States the Atomic Energy Commission and the Geological Survey have prepared a 123-page, pocket-sized booklet entitled, *Prospecting for Uranium*, which may be purchased for 30¢ from the U. S. Government Printing Office, Washington 25, D. C. Orders of 100 or more copies receive a 25% discount. The seven chapters are:

1. The uranium-bearing minerals.
2. Where to look for uranium
3. Testing for uranium
4. Prospecting with the Geiger counter
5. Laboratory assays and selling procedures
6. Laws and regulations
7. Questions frequently asked

In Chapter one, Tables one and two summarize the properties of 20 uranium and thorium minerals listed under the color headings, black, brown, yellow, and green. Uranium detecting methods and devices recommended in Chapter 3 are the fluorescent bead test with LiF or NaF, photographic, scintillation and electroscope tests, and the comparison test by means of a Geiger counter and a sample of known uranium content.

The Atomic Energy Act provides that all uranium and thorium on public lands that were vacant and unappropriated as of August 1, 1946, is reserved "for the use of the United States." This does not prevent prospectors from staking a valid claim on newly found radioactive mineral deposits. However, the Atomic Energy Commission has the right of entry to the claim and the right of removal of the uranium or thorium *without compensation for either metal*. This is to be construed as an emergency provision only, and normally the Commission follows the policy of acquiring ores and concentrates through ordinary commercial methods. Until April 11, 1958, a guaranteed minimum price of \$3.50 per pound of U_3O_8 in domestic ores or concentrates assaying at least 10% U_3O_8 has been established. The Commission will also pay a bonus of \$10,000, until the same date, for the discovery of a new deposit and the production from it of the first 20 short tons of uranium ore or concentrate assaying 20% or more U_3O_8 . *Neither this price nor the bonus will be permitted to apply to the carnotite or roscoelite type of ore*, the purchase of which is governed by a series of labyrinthian schedules printed in full in Appendix 3. Licenses issued by the Commission are required to transfer, deliver, receive, or export radioactive ore after its removal from the deposit.

Licensing legalisms, regulatory ramifications, and pricing procedures, which consume some 40-odd pages or about one-third of the volume, are couched in the usual stupefying governmental jargon, destined no doubt, to discourage the average prospector.

The most valuable part of the book to prospectors will probably be Chapter 7, which answers, in clear, non-technical language 17 basic questions that beginners are likely to ask.

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NEW MINERAL NAMES

Cymrite

W. CAMPBELL SMITH, F. A. BANNISTER AND MAX H. HEY, Cymrite, a new barium mineral from the Benallt manganese mine, Rhiw, Carnarvonshire: *Mineral. Mag.*, **28**, 676-681 (1949).

CHEMICAL PROPERTIES: Analyses were made by Hey, No. 1 below on 7 mg., almost free from impurities; No. 2 on 20 mg. that contained some reddish-brown oxide minerals. No. 3 is the theoretical composition for $\text{BaAlSi}_3\text{O}_8(\text{OH})$.

	SiO_2	Al_2O_3	BaO	H_2O	Fe_2O_3	MnO	Sum
1.	44.8	10.5	[38.5]	3.1	2.8	0.3	[100]
2.	37.65	14.94	31.50	5.31	9.26	0.86	99.52
3.	45.79	12.95	38.97	2.29	—	—	100.00

Heated in a closed tube, the mineral decrepitates, gives off water, and becomes pearly, white, and opaque.

CRYSTALLOGRAPHY: Rotation and Laue photographs show the mineral to be hexagonal. There is a pseudo-cell with $a' 5.33$, $c 7.67$ Å.U., containing $\text{Ba}_2\text{AlSi}_3\text{O}_8(\text{OH})$; the true cell has $a = 8a' = 42.6$ Å.U. Cleavage basal, perfect. Prismatic cleavage noted in thin section. Flakes heated to bright redness had $a 5.29$, $c 7.78$ Å.U., but the index of refraction had dropped to 1.523. X-ray powder data are given.

PHYSICAL AND OPTICAL PROPERTIES: Colorless, in plates up to 7 mm. across and about $\frac{1}{2}$ mm. thick. Also in fibrous specimens with satiny luster. Optically uniaxial, negative; two samples gave (Na light):

	ω	ϵ	Birefringence
1.	$1.6225 \pm .001$	$1.6125 \pm .001$	$0.0094 \pm .0004$
2.	1.6195	1.6115 to 1.6140	0.008 (white light)

For No. 2, ω (Hg-green) 1.624

$D_4^{163.413 \pm .005}$ by suspension in Clerici solution on four carefully selected fragments.

OCCURRENCE: Associated with ganophyllite at the Benallt mine.

NAME: From the Welsh name for Wales, Cymru (pronounced kumry).

MICHAEL FLEISCHER

Llallagualite

MARK C. BANDY, Mineralogia de Llallagua, Bolivia. La Paz, 1946, 69 pp.; through *Mineral. Mag.*, **28**, 732 (1949).

Provisional name for rhombohedral phosphate, which has perhaps the composition of monazite, named for the locality.

M. F.

Patiñoite

M. C. BANDY, *op. cit.*; through *Mineral. Mag.*, **28**, 735 (1949).

"Provisional name for yellow tetragonal crystals, probably a phosphate or arsenate.

Named for Simon Iturbi Patiño, who was the first to work the Lllallagua mines."

DISCUSSION: Names such as these, published without descriptions, have no standing.
M. F.

DISCREDITED MINERALS

Franquenite = Slavikite

RENÉ VAN TASSEL, L'identité entre slavikite et franquenite: *Bull. inst. roy. sci. nat. Belg.*, **25**, No. 7, 15 pp. (1949).

Comparison of franquenite (see *Am. Mineral.*, **31**, 327 (1946)) with slavikite from Valachov, Mandat, and Troja, Czechoslovakia, and Alcaparossa, Argentina, shows their identity. Analyses, optical data, and x-ray data are given.

M. F.

Renierite

J. F. VAES, La Reniérîte (Anciennement appelée "Bornite orange"). Un sulfure germanifère provenant de la Mine Prince-Leopold, Kipushi (Congo belge): *Ann. soc. belge géol.*, *Bull.* **72**, Nos. 1-2, 19-32 (1948).

Re-examination of a mineral previously identified by various workers as orange bornite, cubanite, luzonite, and valleriite showed it to contain germanium.

CHEMICAL PROPERTIES: Analyses of four samples gave

	1	2	3	4
Cu	41.35	41.63	42.05	41.10
Fe	13.84	13.73	13.78	13.73
Ge	6.80	7.75	6.37	6.80
Zn	3.53	3.53	3.94	3.70
As	0.95	0.87	0.79	1.00
S	31.83	31.51	31.69	31.65
Pb	tr.	tr.	tr.	tr.
Sum	98.30	99.02	98.62	98.37 ^a

^a Also Ga tr., Sn 0.16, Insol. 0.20.

Spectrographic analysis showed no element present that would account for the low summations. Soluble in HNO₃.

CRYSTALLOGRAPHIC AND PHYSICAL PROPERTIES: Occurs in idiomorphic grains and in crystals up to 1.5 mm. in geodes. Isometric hextetrahedral, but shows anisotropism in reflected light. Color orange bronze. Hardness 4½, G. higher than that of malachite, lower than that of barite hence about 4.4. An impure sample had G. 4.31. Magnetic and has polarity.

OCCURRENCE: Occurs as inclusions in chalcopyrite, sphalerite, galena, and tennantite, and contains inclusions of tennantite.

NAME: For A. Renier, director of the Geological Survey of Belgium.

DISCUSSION: A comparison is given of renierite and germanite with the conclusion that the differences in chemical composition and physical properties are too great to consider renierite a variety of germanite. The differences are 13.8% Fe, (7.8% in germanite), bronze in color (germanite is reddish-gray), magnetism (not stated whether germanite is magnetic), anisotropy, (but it is stated that this may be an anomaly and that x-ray study is needed).

It seems to me that until further evidence is provided, the name reinierite is unjustified for what appears to be ferroan germanite.

M. F.